NATIONAL BUREAU OF STANDARDS

NBS SPECIAL PUBLICATION 260-71

U.S. DEPARTMENT OF COMMERCE / National Bureau of Standards

Standard Reference Materials:

SUMMARY OF THE CLINICAL LABORATORY STANDARDS ISSUED BY THE NATIONAL BUREAU OF STANDARDS

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NBS special publication

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Preface

Standard Reference Materials (SRM's) as defined by the National Bureau of Standards are well-characterized materials, produced in quantity and certified for one or more physical or chemical properties. They are used to assure the accuracy and compatibility of measurements throughout the Nation. SRM's are widely used as primary standards in many diverse fields in science, industry, and technology, both within the United States and throughout the world. They are also used extensively in the fields of environmental and clinical analysis. In many applications, traceability of quality control and measurement processes to the national measurement system is carried out through the mechanism and use of SRM's. For many of the Nation's scientists and technologists it is therefore of more than passing interest to know the details of the measurements made at NBS in arriving at the certified values of the SRM's produced. An NBS series of papers, of which this publication is a member, called the NBS Special Publication – 260 Series, is reserved for this purpose.

This 260 Series is dedicated to the dissemination of information on different phases of the preparation, measurement, certification and use of NBS-SRM's. In general, much more detail will be found in these papers than is generally allowed, or desirable, in scientific journal articles. This enables the user to assess the validity and accuracy of the measurement processes employed, to judge the statistical analysis, and to learn details of techniques and methods utilized for work entailing the greatest care and accuracy. These papers also should provide sufficient additional information not found on the certificate so that new applications in diverse fields not foreseen at the time the SRM was originally issued will be sought and found.

Inquiries concerning the technical content of this paper should be directed to the author(s). Other questions concerned with the availability, delivery, price, and so forth, will receive prompt attention from:

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National Bureau of Standards
Washington, D. C. 20234

George A. Uriano, Chief
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TABLE OF CONTENTS

Abstract

This publication is a summary of the Clinical Laboratory Standards issued by NBS as Standard Reference Materials (SRM's). The material, composition, certification, use, and remarks concerning each of the SRM's described are presented in tabular form. Copies of the certificates for these SRM's are contained in the appendix for more detailed information.

Key Words: Chemical composition; clinical materials; Standard Reference Materials.

Introduction

Since its inauguration in 1901, the National Bureau of Standards (NBS) has issued nearly 2000 different Standard Samples or Standard Reference Materials (SRM's). Many of these have been renewed several times, many have been replaced or discontinued as technology changed. Today, over 1000 SRM's are available, together with a large number of scientific publications related to the fundamental and applied characteristics of these materials. Each material is certified for chemical composition, chemical properties, or its physical or mechanical characteristics. Each SRM is provided with a Certificate or Certificate of Analysis that contains the essential data concerning its properties or characteristics. The SRM's currently available cover a wide range of chemical, physical, and mechanical properties, and a corresponding wide range of measurement interests in practically all aspects of fundamental and applied science. These SRM's constitute a unique and invaluable means of transferring to the user accurate data obtained at NBS, and provide essential tools that can be used to improve accuracy in practically all areas where measurements are performed.

In addition to SRM's, the National Bureau of Standards issues a variety of Research Materials (RM's) having various properties described in individual "Reports of Investigation". They are intended primarily to further the scientific or technical research on that particular material. Other materials, called Special Reference Materials (GM's), are also available from NBS. These are materials, produced and certified by other Government agencies, standard organizations, or other nonprofit organizations, that are considered useful to the public and for which no alternate method of national distribution exists.

The various categories of materials available from NBS are given in Table 1. This lists these materials according to their chemical composition, physical properties, or engineering characteristics. A more detailed alphabetic enumeration of these materials is given in Appendix I. Table 1 and Appendix I were taken from NBS Special Publication 260, NBS Standard Reference Materials Catalog, 1979-80 Edition[2]. This publication lists every material available from the NBS Office of Standard Reference Materials.

Further information on the reference materials available from NBS may be obtained from the Office of Standard Reference Materials, National Bureau of Standards, Washington, D. C. 20234. Information on other NBS services may be obtained from the Technical Information and Publications Division, National Bureau of Standards, Washington, D. C. 20234.

In addition to these types of materials, NBS provides many additional services. These include: Measurement Assurance Programs, Calibration and Related Measurement Services, Proficiency Sample Programs, a National Voluntary Laboratory Accreditation Program, Standards Information Services, Standard Reference Data, and Technical Information and Publications.

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[2]For sale by the Superintendent of Documents, U. S. Government Printing Office, Washington, D. C. 20402, under Stock No. 003-003-20248. (Price $3.00, add 25 percent for foreign orders.)

Table 1. Categories of Standard Reference Materials available from the National Bureau of Standards.

CERTIFIED CHEMICAL COMPOSITION STANDARDS

Steels (chip form)

- Plain carbon
- Low alloy
- High alloy
- Stainless
- Tool

Steels (granular form)

Steels (solid form)

- Ingot iron and low alloy
- Special ingot irons and low alloy
- Stainless
- Specialty
- High-temperature alloys
- Tool

Steelmaking Alloys

Cast Irons (chip form)

Cast Steels, White Cast Irons, Ductile Irons, and Blast Furnace Irons (solid form)

Nonferrous Alloys (chip form)

- Aluminum "Benchmarks"
- Cobalt
- Copper
- Copper "Benchmarks"
- Lead
- Magnesium
- Nickel
- Nickel Superalloy, Trace Elements
- Nickel oxide
- Selenium
- Tin
- Titanium
- Zinc
- Zirconium

Nonferrous Alloys (solid form)

- Aluminum "Benchmarks"
- Copper
- Copper "Benchmarks"
- Lead
- Nickel
- Titanium
- Zinc
- Zirconium

Gases in Metals

High-Purity Metals

Electron Probe Microanalytical Standards

Primary, Working, and Secondary Standard Chemicals

Microchemical Standards

Clinical Laboratory Standards

Biological Standards

Environmental Standards

- Analyzed gases
- Analyzed liquids and solids
- Permeation tubes

Industrial Hygiene Standards

Forensic Standards

Hydrocarbon Blends

Metallo-Organic Compounds

Fertilizers

Ores

Minerals, Refractories, Glasses, and Carbides

Cement

Trace Element Standards

Nuclear Materials

- Special nuclear materials
  - Plutonium assay
  - Plutonium isotopic
  - Uranium assay
  - Uranium isotopic
- Neutron density standards
- Fission track glass standards

Isotopic Reference Standards

Table 1. continued.

CERTIFIED PHYSICAL PROPERTY STANDARDS

Ion Activity Standards

- pH standards
- pD standards
- Ion selective electrodes

Mechanical and Metrology Standards

- Magnification
- Coating thickness
- Glass
- Elasticity
- Density
- Polymer
- Rheology

Heat Standards

- Superconductive thermometric fixed point devices
- Freezing points
  - Defining fixed points
  - Determined reference points
- Melting points
- Calorimetric
  - Combustion
  - Solution
  - Heat source
  - Enthalpy and heat capacity
- Vapor pressure
- Thermal expansion
- Thermocouple materials
- Thermal resistance

Magnetic Standards

- Magnetic susceptibility
- Magnetic moment
- Paramagnetic resonance

Optical Standards

- Spectrophotometric
- Thermal emittance
- Refractive index

Radioactivity Standards

- Alpha-particle standards
- Beta-particle and gamma-ray gas standards
- Alpha-particle, beta-particle, gamma-ray, and electron-capture solution standards
- Contemporary standard for carbon-14 dating laboratories
- Environmental standards
- Low energy photon sources
- Gamma-ray "point-source" standards
- Radium gamma-ray solution standards
- Radium solution standards for randon analysis
- Radioactivity standard reference materials currently not in stock

Metallurgical

Mössbauer

X-ray Diffraction

Gas Transmission

Permittivity

Reference Fuels

Resistivity

ENGINEERING TYPE STANDARDS

Standard Rubber and Rubber-Compounding Materials

Reference Magnetic Tapes

Centerline Drawings, OCR-B

Sizing Standards

- Glass spheres for particle size
- Turbidimetric and fineness (cement)

RESEARCH MATERIALS

Color Standards

X-ray and Photographic Standards

Surface Flammability Standards

Smoke Density Chamber Standards

Water Vapor Permeance

Tape Adhesion Testing Standard

SPECIAL REFERENCE MATERIALS

The first NBS Standard Reference Material designed specifically for clinical laboratory use was issued in December 1967. This standard, SRM 911, Cholesterol, was developed in response to requests from both the College of American Pathologists and the American Association for Clinical Chemistry. In the 12 years since then, the number of clinical SRM's has grown to 30, and has been supplemented by new spectrophotometric and temperature standards that are also useful in the clinical field. During the next decade this growth is expected to continue at much the same rate.

The SRM's produced (and those planned), especially since 1973, have closely followed the priority list for clinical SRM's established in 1973 by the NBS Analytical Chemistry Division[3] Clinical Chemistry Advisory Panel.

This publication is an attempt to describe in general terms the composition, certification, and use of the clinical laboratory SRM's and those other SRM's especially useful in such laboratories. No attempt has been made, however, to include all of the SRM's that may be used in clinical laboratories, e.g., primary chemicals, pH standards, etc., nor has an attempt been made to list other possible uses for these materials. Many of them would have applications in general analytical laboratories and in laboratories where the accuracy of molecular absorption spectrometry is of primary interest.

Table 2 contains the essential information concerning the material composition, the certification parameters, and use. Under "Remarks," additional data such as storage conditions and stability is provided.[4] All the data and information contained in this table were extracted from the Certificates or Certificates of Analysis issued for the SRM's included in the table, except those SRM's listed as "In Preparation". An examination of this table gives the reader a general view of these SRM's. For more detailed information, the individual Certificates reproduced in Appendix II should be consulted as well as the references cited in each Certificate.

In the table, similar types of SRM's are grouped together to facilitate comparisons of their properties. This should prove especially useful for the spectrophotometric SRM's where both wavelength range and material composition are critical to the user. The Certificates in Appendix II, however, are arranged in numerical order. The SRM's listed in the table include all of the clinical laboratory standards that were issued or were in preparation by the end of 1979. These SRM's are the result of the concerted efforts of more than 70 scientists from the NBS National Measurement Laboratory. Each Certificate lists the individuals who contributed to the certification of the SRM.

In addition to the SRM's and their Certificates, NBS issues a series of Special Publications (SP), called the "260 Series," that relate directly to Standard Reference Materials as stated in the preface. The list of available publications in the "260 Series" is given at the beginning of this publication.[5]

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[3]Now the Center for Analytical Chemistry.

[4]NOTE: The use of proprietary designations in Table 2 is for information only, and should not be construed as an endorsement of the product by either the Department of Commerce or the National Bureau of Standards.

[5]For complete bibliographic reference and ordering information, see "Other Publications in this Series," pp. VII-X.

Several of these publications describe the certification or use of SRM's listed in Table 2, and several others deal with methodology important to clinical laboratories. Table 3 matches the publication number to the related SRM number and gives an abbreviated title for the publication. Planned publications in this series of clinical interest are also listed in the table.

Table 2. Summary of Clinical Laboratory Standards

A. Organics

SRM	Material	Composition
900 Antiepliepsy Drug Level Assay Standard	A mixture of 4 antiepilepsy drugs in a processed human serum base. The material was processed, vialed, and packaged by Microbiological Associates, Walkersville, Maryland.	SRM 900 contains concentration levels of the 4 drugs at the toxic, therapeutic, and subtherapeutic levels, in µg/mL for phenytoin ethosuximide, phenobarbital, and primidone, respectively, are: 60.7, 16.7, and 4.2; 174.7, 75.9, and 11.8; 103.6, 21.6, and 5.3; and 18.6, 8.1, and 3.6.
909 Human Serum	In preparation at time of writing. Certificate will be included in Appendix B if available at time of publication.	
910 Sodium Pyruvate	In preparation at time of writing. Certificate will be included in Appendix B if available at time of publication.	
911a Cholesterol	Cholesterol of known purity. The material was obtained from J. T. Baker Chemical Company, Phillipsburg, New Jersey.	Purity is 99.8%. Test for lathosterol is negative. Melting point is 149.0 to 149.4 °C. Specific rotation $[a]_D^{20}$ is -0.700 rad (-40.1°) (c,1; $CHCl_3$). Carbon 83.86±0.05 wt.%; hydrogen 12.00±0.05 wt.%. Loss at 100 °C and 0.02 torr is 0.01% per hour over 1.5 day period.
912a Urea	Urea of known purity. The material was obtained from Fluka AG, Switzerland	Purity is 99.9%; moisture is 0.02%; biuret is 0.02%; ash is 0.001%; insoluble matter is 0.0001. The inaccuracy for the value of purity is estimated to be 0.1%. Melting range is 133.0 to 134.0 °C. A 10% aqueous solution of urea showed a pH of 7.1±0.2 at 23 °C.

Certification	Use	Remarks
The analytical techniques used in the certification were liquid and gas chromatography. Immunoenzymatic assays have also been used. The certification uncertainties, at one standard error, were from ±0.1 to ±0.9 µ/mL according to the compound and the concentration.	For the calibration of clincal procedures for the determination of these 4 components in serum and for the evaluation of reference solutions prepared in house or supplied commercially. For "in vitro" diagnostic use only. For use, the materials should be reconstituted with high-purity water, and this solution used within 1 day.	SRM 900 must be handled as if capable of transmitting hepatitis. It is supplied in a set of 4 individual freeze-dried preparations that should be stored at 4 °C in the dark. Under these conditions the material is stable for 2 years.
The purity was determined by liquid chromatography; gravimetric recovery; thin-layer chromatography; gas chromatography; neutron activation; infrared and ultraviolet absorption spectrometry. Two impurities were found representing a combined weight of 0.08% of the initial sample. Bromine content is 12 µg/g; chlorine content is 0.7 µg/g; iodine content is less than 0.8 µg/g ±20%.	For the calibration and standardization of procedures for cholesterol determinations in clinical analysis and for evaluation of daily working standards. For "in vitro" diagnostic use only. Stock standard solutions are prepared by dissolving in absolute ethanol (5 mmol/L). The solutions should be clear.	Cholesterol should be stored in tightly closed bottles in a refrigerator or freezer, protected from sun or ultraviolet radiation. When stored under inert gas, as delivered, and at -15 °C the material is stable for 10 years. If stored in the dark at room temperature the material should not be used after 6 months from the date of purchase. The 5-mmol/L ethanol solution at 0 °C is stable for 4 months; the 2.5-mmol/L acetic acid solution at room temperature is stable for 3 months.
The apparent purity was determined by differential scanning calorimetry. Biuret was estimated spectrophotometrically. Moisture was determined by the Karl Fischer titration.	For the calibration and standardization of procedures for urea nitrogen determinations in clinical analysis and for evaluation of daily working standards. For "in vitro" diagnostic use only. A standard solution containing 20 mg/100 mL of urea nitrogen is prepared from 0.429 g urea in 1000 mL of ammonia-free distilled water.	Urea should be stored in its tightly closed container to prevent change in the moisture content. It should not be exposed to heat, moisture, or direct sunlight. Refrigerated storage is recommended. Under proper storage urea is stable for at least 5 years. The standard urea nitrogen solution (20 mg/100 mL) is stable for 3 months when stored at 4 °C in a well-stoppered, all-glass container.

Table 2. A. Organics - continued.

SRM	Material	Composition
913 Uric Acid	Uric acid of known purity. The material was obtained from the Pfanstiehl Laboratories, Inc., Waukegan, Illinois.	Purity is 99.7%; volatile matter is 0.14%; ash is 0.057%. Purity is given with an estimated inaccuracy of 0.1%. The ash contains: Na, K, Al, Ca, Fe, P, Si, Co, Cu, Mn, Ni, Zn.
914 Creatinine	Creatinine of known purity. The material was obtained from the Pfanstiehl Laboratories, Inc., Waukegan, Illinois.	Purity is 99.8%; volatile matter is 0.03%; chloride is 0.07%; ash is 0.003%; insoluble matter is 0.001%. The value of purity has an estimated inaccuracy of 0.1%. Emission spectrometric analysis of the ash showed the presence of Si, Al, Na, Ti, Fe, Mg. Neutron activation analysis indicated the presence of Cl, Cu, Mn.
916 Bilirubin	Bilirubin of known purity. The material was obtained from the Pfanstiehl Laboratories, Inc., Waukegan, Illinois. It was prepared from material that was isolated from hog bile and crystalized as the acid. It was purified in chloroform solution with sodium sulfate and recrystalized from chloroform.	Purity is 99.0%; chloroform is 0.8%; insoluble in chloroform less than 0.01%; ash is 0.01%. The value of the bilirubin content has a possible estimated inaccuracy of 2%. Flame emission and atomic absorption measurements show the presence of Li, Na, K, Ca, Mg. Elemental composition: C, 67.36; H, 6.24; N, 9.42; Cl, 0.73.

Certification	Use	Remarks
The purity and homogeneity of the material was established by paper and thin-layer chromatography, emission spectrometry; neutron activation, ultra-violet absorption.	For the calibration and standardization of procedures and evaluating working standards for uric acid determinations in clinical analysis. For "in vitro" diagnostic use only. Standard solutions made from this material should be kept in dark-brown bottles glass-stoppered, and stored at 4 °C.	Uric acid should be stored in well-closed containers at 30 °C or less, and be protected from heat and sunlight. Under these conditions the material is stable for at least 5 years. The stock standard solutions 1 mg/mL are stable for 3 months when stored in a well-stoppered, all-glass, dark-brown bottle at 4 °C.
The homogeneity and purity of the material was established by paper, thin-layer, and gas-liquid chromatography, and by phase-solubility analysis. The chloride was determined by titration of a solution obtained from an oxygen flask combustion. Ultra-violet, infrared, and nuclear magnetic resonance spectra showed no evidence of impurities.	For the calibration and standardization of procedures and the evaluation of working standards for creatinine determinations in clinical analysis. For "in vitro" diagnostic use only. A "stock" standard solution is made by dissolving 0.100 g of creatinine into 100 mL 0.1 N HCl.	Creatinine should be stored in well-closed container at 30 °C or less, protected from heat and sunlight. Refrigerated storage is recommended. Under these conditions the material is stable for 5 years. The "stock" standard solution (1 mg/mL) prepared as described was found to be stable indefinitely when stored at 4 °C in a well-stoppered all-glass container.
The certification of SRM 916 is based on the best current state-of-the-art knowledge. In spite of decades of work on bilirubin, it is still felt that much of its basic chemistry is incomplete.	For the calibration and standardization of procedures for bilirubin determinations for for evaluation of the daily working standards. For "in vitro" diagnostic use only.	Bilirubin is light sensitive, thus all manipulation should be performed under low-intensity incandescent light. It should be stored under conditions that totally exclude light. SRM 916 should be kept in the tightly closed vial in a desiccator at 4 °C. Under proper storage the material is stable for at least 3 years. Solutions of bilirubin prepared as described in the Certificate may be preserved for about 1 week at -20 °C.

Table 2. A. Organics - continued.

SRM	Material	Composition
917 D-Glucose (Dextrose)	D-Glucose of known purity. The material was obtained from the Pfanstiehl Laboratories, Inc., Waukegan, Illinois.	Purity is 99.9%; α-D-Glucopyranose >99.0%; β-D-Glucopyranose <1.0%; moisture 0.06%; ash 0.002%. Insoluble matter 0.001 to 0.006% nitrogen <0.001%. Purity is given with an estimated inaccuracy of ±0.1%. Paper, thin-layer, and high pressure ion-exchange chromatography revealed no organic impurities. Emission spectrometry showed the presence of Ca, Mg, Si, Al, B, Fe, Cu, Na. Turbidimetry showed the presence of S, Cl.
920 D-Mannitol	D-Mannitol of known purity. The material was obtained from the Pfanstiehl Laboratories, Inc., Waukegan, Illinois.	Purity is 99.8%; D-Glucitol is 0.1%; total Alditol is 99.9%; loss on drying is <0.02%; ash is <0.001%; insoluble matter is <0.001%. The value for purity has an estimated inaccuracy of 0.1%. Melting point is 167.0-168.0 °C. Carbon 39.51% and hydrogen 7.86%.
921 Cortisol (hydrocortisone)	Cortisol of known purity. The material was obtained from the Upjohn Co., Kalamazoo, Michigan.	Purity is 98.9%; 21-Dehydrocortisol is 0.6%; 21-Ω-Acetylcortisol is 0.2%; 21-Dehydrocortisone is 0.1%; Cortisone is 0.1%; total steroids is 99.9%; ash is 0.002%; insoluble matter is 0.001%; loss on drying is 0.08%. The cortisol assay has an estimated inaccuracy of 0.2%. Elemental analysis showed carbon 69.49% and hydrogen 8.39%. Melting point is 219.0 to 220.5 °C.

Certification	Use	Remarks
The proportion of α-D-Glucose anomer was estimated by gas-liquid chromatography, differential scanning calorimetry, and proton magnetic resonance spectroscopy at 90 MHz.	For calibration and standardization of procedures for glucose determinations and evaluation of working standards in clinical analysis. For "in vitro" diagnostic use only. All solutions of D-Glucose should be clear and without bacterial growth.	D-Glucose should be stored in a well-closed container at 30 °C or less, protected from heat or direct sun-light. Refrigerated storage is recommended. Under proper storage this material is stable for at least 5 years.
The purity of the material was determined by differential scanning calorimetry; phase solubility analysis; thin-layer chromatography; infrared and ultraviolet spectrometry; and proton and carbon-13 magnetic resonance spectroscopy. Optical rotation in water is $[\alpha]_D^{20} = -0.3°$.	For calibration and standardization of procedures for determination of triglycerides in clinical chemistry. For "in vitro" diagnostic use only.	The material should be stored in a well-closed container at 30 °C or less, protected from direct sun-light. Under these conditions it is stable for at least 5 years. Stock standard solutions (1 µmol/mL) should be stored in glass stoppered, brown bottles at 4 °C. Under such conditions these standards should be stable for 6 months.
The identification and quantitation of the 4 steroid impurities were made by Fourier-transform proton-magnetic resonance spectroscopy; liquid and thin-layer chromatography. Homogeneity was established by C and H analysis. Optical rotation at λ 589 nm is 168.9°, and at λ 365 nm is 571.7° at 20 °C.	For the calibration and standardization of procedures for cortisol determinations in clinical analysis. For "in vitro" diagnostic use only.	The material should be stored in a well-closed container at 30 °C or less, protected from direct sun-light. Refrigerated storage is recommended. Under these conditions the material is stable for 5 years. The solution of 1 mg/mL cortisol in ethanol should be stored in a well-stoppered, all-glass container at 4 °C. Under these conditions the solutions are stable for 6 months.

Table 2. A. Organics - continued.

SRM	Material	Composition
925 4-Hydroxy-3-Methoxy-DL-Mandelic Acid (VMA)	VMA of known purity. The material was prepared at NBS.	Purity is 99.4%; uncharacterized compound 0.5%; keto-VMA 0.1%; volatile matter 0.02%; ash 0.004%. The value of purity has an estimated inaccuracy of 0.4%. Melting range is 133-134 °C. Elemental analysis: carbon 54.6; hydrogen 5.1; oxygen 40.3.
926 Bovine Serum Albumin (Total Protein Standard)	Bovine Serum Albumin of known purity. It was prepared by the Research Division, Miles Laboratories, Inc. Kankakee, Illinois.	Mass fractions (g/g): Ash 0.0006; hexose 0.00034; citrate + pyruvate + lactate 0.00015; lipid 0.0003; non-protein amino compound 0.00002; water 0.048; protein impurities (dimer) by: gel electrophoresis, 0.043; gel chromatography, 0.050.
927 Bovine Serum Albumin (7% Solution) (Total Protein Standard)	Solution of known protein concentration and purity prepared from SRM 926 described above. It was prepared by the Research Division, Miles Laboratories Inc., Kankakee, Illinois.	The 7% solution was prepared from SRM 926. The aqueous solution is brought to proper ionic strength with NaCl and to proper pH with NaOH. It was sterilized by membrane filtration.
938 Para-Nitrophenol	In preparation at time of writing. Certificate will be included in Appendix B if available at time of publication.	

B. Organo-Metallic

929 Magnesium Gluconate Dihydrate (Clinical Standard for Magnesium)	Magnesium gluconate dihydrate of known purity. This material was obtained from the J. T. Baker Chemical Co., Phillipsburg, New Jersey	Practically pure magnesium gluconate dihydrate.

Certification	Use	Remarks
Thin-layer chromatography has revealed two impurities. Phase solubility analysis of VMA were inconclusive. The sample contains a carboxylic acid equivalent of 99.83%. The material was characterized by gas-liquid chromatography.	For calibration and standardization of procedures for VMA used in clinical analysis. For "in vitro" diagnostic use only.	Solutions of VMA exposed to light and air are unstable. VMA should be kept in the well-closed original bottle and in a desiccator. Refrigeration at 4 °C, or better, -20 °C is recommended. VMA should be protected from sunlight or ultraviolet radiation. The material should not be used after 5 years from the date of purchase.
SRM 926 conforms to the specifications approved by the National Committee for Clinical Laboratory Standards. The analytical techniques used in the certification of this material are those recommended by the National Committee.	For calibration of procedures for total serum protein determinations. For "in vitro" diagnostic use only.	SRM 926 should be stored in a well-closed container at 4 °C, protected from sunlight or ultraviolet radiation. Under these conditions it is stable for 3 years. The lyophilized material is extremely hygroscopic.
SRM 927 conforms to the specification for standardized protein solution approved by the National Committee for Clinical Laboratory Standards. Peptide mass (g/L) 70.45 (biuret method); 70.48 (Kjeldhal method) 70.77 (optical density method); pH 6.66.	For calibration of procedures for total serum protein determinations and for "in vitro" diagnostic use only. SRM 927 should not be used in dye-binding tests, for checking refractometers, or as an immunochemical standard. Not recommended for use in bilirubin tests.	The solution is supplied in sealed ampoules and must be stored at 4 °C. Opened ampoules should not be stored. Sealed ampoules stored at 4 °C are stable for 3 years. SRM 927 should not be used after 3 years from purchase. Once an ampoule is opened, the solution should be used promptly.
Certified for magnesium content of 5.403 wt. %, by thermal ionization isotope dilution mass spectrometry.	For calibration and standardization of clinical procedures for the determination of magnesium. For "in vitro" diagnostic use only.	Magnesium gluconate should be stored in the tightly-closed, original bottle at room temperature. It is hygroscopic and must be dried before use. Under proper storage, the material should be stable for 5 years. Solutions prepared from SRM 929 are stable under normal laboratory conditions for at least 60 days.

Table 2. C. Inorganic

SRM	Material	Composition
915 Calcium Carbonate	Calcium carbonate of known purity. The material was obtained from the J. T. Baker Chemical Co., Phillipsburg, New Jersey.	Purity is 99.9+%; water is 0.01%. Emission and atomic absorption spectrometry, neutron activation analysis, and spectrophotometry were used to determine the following trace metals: Cu, Fe, Mg, Mn, Si, Na, Sr, K, Li, Ba.
918 Potassium Chloride (Clinical Standard)	Potassium chloride of known purity. The material was obtained from the J. T. Baker Chemical Co., Phillipsburg, New Jersey.	Purity is 99.9%. Moisture is 0.07%. Emission spectrometry and atomic absorption showed the presence of traces of Al, Cu, Fe, Mg, Rb, Na, Li, Cs, Ca.
919 Sodium Chloride (Clinical Standard)	Sodium chloride of known purity. The material was obtained from J. T. Baker Chemical Co., Phillipsburg, New Jersey.	Purity is 99.9%. Moisture is about 0.08%. Emission spectrometry and atomic absorption showed the presence of traces of Ca, Cu, Fe, Mg, K, Cs, Rb, Li.
924 Lithium Carbonate	Lithium carbonate of known purity. The material was obtained from J. T. Baker Chemical Co., Phillipsburg, New Jersey.	Purity is 100.0_5%; based on coulometric acidimetry. The molecular weight is 73.9486 based on the mass-spectrometrically determined value of 6.9696 for the atomic weight of lithium. Emission spectroscopy test for trace metals was negative. Flame emission and atomic absorption showed that alkali metals are below 1 ppm and calcium is 4 ppm.

Certification	Use	Remarks
Material meets or exceeds the minimum requirements specified for reagent grade calcium carbonate as given in Reagent Chemicals published by the American Chemical Society. The compound is stable up to 625 °C, above which decomposition occurs as indicated by thermal gravimetric analysis.	For the calibration and standardization of procedures for calicium determinations in clinical analysis. For "in vitro" only diagnostic use. SRM 915 may be used to prepare Ca standard solution for flame emission, atomic absorption, or titrimetric methods of analysis. (See, NBS Special Publication 260-36.)	Calcium Carbonate should be stored in the tightly-closed original bottle at room temperature. Under proper storage this material was found to be stable for at least 10 years. It is recommended that the material not be used after 5 years from the date of purchase. Solutions prepared from SRM 915 are stable indefinitely when stored in glass-stoppered bottles. All solutions should be clear.
The material meets or exceeds the specifications for reagent grade potassium chloride as given in Reagent Chemicals published by the American Chemical Society. Potassium Chloride is homogeneous and was assayed at 99.98%.	For use as a standard for potassium and, to a lesser degree, for chloride determinations in clinical chemistry. (See, NBS Special Publications 260-63 and 260-67.) For "in vitro" diagnostic use only.	Potassium Chloride should be stored in the well-closed original container under normal laboratory conditions. The weighing and other manipulations should not be made when the relative humidity exceeds 75%. The solutions of SRM 918 are stable indefinitely when stored in a well-stoppered, all-glass container.
The material meets or exceeds the specifications for reagent grade sodium chloride as given in Reagent Chemicals published by the American Chemical Society. Coulometric determination of Cl indicate a purity of 99.995%.	For use as a standard for the determination of sodium and chlorine ions in clinical chemistry. (See NBS Special Publications 260-60 and 260-67.) For "in vitro" diagnostic use only.	Sodium chloride should be stored in the well-closed original bottle under normal laboratory conditions. The weighing and other manipulations should not be made where the relative humidity exceeds 60%. Solutions of SRM 919 are stable indefinitely when stored in a well-stoppered, all-glass container.
No actual lithium determination was made. The certification is based on the analysis of the carbonate anion and the proven absence of metallic cations above trace levels. The material meets or exceeds the specifications for reagent grade lithium carbonate as given in Reagent Chemicals published by the American Chemical Society.	For calibration and standardization of procedures for the determination of lithium in clinical chemistry. For "in vitro" diagnostic use only.	SRM 924 should be kept in the well-closed original bottle under normal laboratory conditions. Solutions of this material are stable indefinitely when stored in a well-stoppered, all-glass container. The solutions should be clear.

Table 2. C. Inorganic - continued.

SRM	Material	Composition
928 Lead Nitrate (Clinical Standard)	Lead nitrate of known purity. The material was obtained from J. T. Baker Chemical Co., Phillipsburg, New Jersey.	Assay is 100.00%. Molecular weight is 331.219 based on a mass-spectrometric determination of 207.209 for the atomic weight of lead in the sample. Emission spectroscopy showed the presence of Ag, Cr, Ni.
937 Iron Metal (Clinical Standard)	Electrolytic iron metal of known purity. The material was taken from a selected lot of high-purity iron available at NBS.	Assay is 99.90 wt. %. Impurities are: Ni, Si, C, Cr, Co, Cu, Mn, O, S, Mo, P, Ge, N. Other elements are <0.003%.

D. Ion-Activity

922 Tris(Hydroxymethyl)-Aminomethane and 923 Tris(Hydroxymethyl)-Aminomethane Hydrochloride	Compounds of known purity. The materials were obtained from Sigma Chemical Co., St. Louis Missouri.	Purity is 99.99 and 99.69 mole percent, respectively.
2203 Potassium Fluoride	Potassium fluoride of reagent-grade purity.	After drying, the KF assays at 99.5%. It is not to be considered entirely free of traces of chloride, fluorosilicates, and heavy metals.

Certification	Use	Remarks
The material meets or exceeds the specifications for reagent grade lead nitrate as given in Reagent Chemicals, published by the American Chemical Society.	For calibration and standardization of procedures for lead determination in clinical chemistry. For "in vitro" diagnostic use only.	This material should be stored in the tightly-closed original bottle under normal laboratory conditions. The material will not absorb appreciable water when exposed to 90% relative humidity atmosphere for 5 days.
The assay was performed by dissolution in hydrochloric acid and by reduction with stannous chloride and oxidation of the excess with mercuric chloride.	For calibration and standardization of procedures for iron determination in clinical chemistry. For "in vitro" diagnostic use only.	SRM 937 should be stored in the tightly-closed bottle under normal laboratory conditions.
The purity of the materials was established by coulometry. The solution used for calibration of pH equipment is 0.01667 molal (SRM 922) and 0.0500 molal (SRM 923). The pH(S) of the solution at 20 °C is 7.840.	For secondary standard pH buffer solutions in clinical chemistry, and in cases where phosphate buffers would cause undesirable side reactions. The buffer solution is sensitive to carbon dioxide. For "in vitro" diagnostic use only.	SRM 922 should be stored in a well-closed container at room temperature and protected from sunlight. Under these conditions the material is stable for 12 years. SRM 923 should be stored in similar conditions. It is stable for at least 5 years. SRM 923 is hygroscopic and should not be manipulated in relative humidity exceeding 50%. Neither SRM 922 nor SRM 923 should be used after 5 years from date of purchase.
Solutions of the KF are certified for the activity coefficient of the fluoride ion and the related pF value at 25 °C over the molality range of 0.0001 to 2.0.	For the standardization of fluoride ion-selective electrodes.	SRM 2203 is hydroscopic and must be dried in two stages before use: 2 hours at 110 °C followed by 2 hours at 200±10 °C.

Table 2. E. Temperature

SRM	Material	Composition
933 Clinical Laboratory Thermometers	Set of three thermometers. The thermometers were manufactured by Princo Instruments, Inc., Southampton, Pennsylvania.	This SRM consists of three individually calibrated thermometers. Each unit is a solid-stem, mercury-in-glass instrument 180 mm in length marked with a 95 mm immersion line. Each is nitrogen filled above the mercury. The main scales of the thermometers are 24.00 to 26.00 °C; 29.00 to 31.00 °C; and 36.00 to 38.00 °C respectively with 0.05 °C divisions.
934 Clinical Laboratory Thermometer	One thermometer manufactured by Princo Instruments, Inc., Southampton, Pennsylvania..	The thermometer is a solid-stem, mercury-in-glass instrument 300±5 mm long, and marked with a 95-mm immersion line. Nitrogen fills the space above the mercury. The main scale extends from 24.00 to 38.00 °C in 0.05 °C divisions.
1968 Gallium Melting-Point Standard	Gallium metal of high-purity obtained from Eagle-Picher Industries, Inc., Quapaw, Oklahoma.	The nominal purity of the material is 99.99999%. SRM 1968 is contained in a specially designed epoxy-sealed cell made at NBS from a Teflon body, a nylon well, and a nylon cap stem.

F. Spectrophotometry and Fluorescence

1. Solids

SRM	Material	Composition
930 Glass Filters for Spectrophotometry	Solid filters made of Schott NG 4 and NG 5 optically neutral glass. Obtained from the Schott-Jenaer Glaswerk, Mainz, Germany,	Three filters with nominal transmittances of 10, 20, 30%. Each filter is mounted in a black anodized aluminum holder provided with front and rear shutters. The filters are stored in a cylindrical aluminum container.

Certification	Use	Remarks
Calibrated points are 0 °C and 25, 30, or 37 °C depending on the scale of the individual thermometer. (See NBS Special Publication 260-48.)	SRM 933 is intended for use in clinical laboratories as a primary calibrant in clinical enzymology.	Each thermometer has an auxiliary scale from -0.20 to +0.20 °C with 0.05 °C divisions.
Calibrated points are 0, 25, 30, and 37 °C. (See NBS Special Publication 260-48.)	SRM 934 is intended for use in clinical laboratories as a primary calibrant in clinical enzymology.	The thermometer has an auxiliary scale from -0.20 to +0.20 °C with 0.05 °C divisions.
The certified melting point is 29.7723 °C. It was established using a set of 3 stable thermistors that were calibrated against a standard Platinum Resistance Thermometer. The uncertainty represents one-half of the total range of the temperatures observed in 152 determinations of the melting points of this and similar samples of SRM 1968.	SRM 1968 is intended to be used to calibrate thermometers near 30 °C. It should be used in a temperature-regulated bath.	SRM 1968 can be used to calibrate all thermometers that have temperature sensing elements smaller than 3.5 mm in diameter. The gallium cell should not be immersed in water for more than 2 days. Water should not enter the cell. (See NBS Special Publication 481, Gallium Melting-point Standard (1977).)
The transmittance of each filter is measured with the high-accuracy spectrophotometer at λ 440, 465, 546.1, 590, 635 nm, using spectral bandpasses of 2.2, 2.7, 6.5, 5.4, 6.0 nm respectively.	This SRM is intended as a reference source for the verification of the transmittance and absorbance scales of conventional spectrophotometers.	The use of this SRM is limited to the visible spectrum and requires narrow spectral bandpasses. For further details consult the Certificate included in the Appendix; see also NBS Special Publication 260-51.

Table 2. F. Spectrophotometry and Fluorescence - continued.

SRM	Material	Composition
2030 Glass Filter for Transmittance Measurements	Solid filters made of Schott NG 4 and NG 5 optically neutral glass. Obtained from the Schott-Jenaer Glaswerk, Mainz, Germany,	One glass filter with a nominal transmittance of 30% in a black anodized aluminum holder with shutters. The filter is stored in a wood-like plastic box.
2031 Metal-on-Quartz Filters for Spectrophotometry	Solid filters made of semitransparent evaporated chromium-on-fused silica (non-fluorescent) plates. Sets 1 through 95 prepared by Cosmo Optics, Inc., Middletown, New York; subsequent sets prepared by NBS Optical Shop.	Three filters with nominal transmittances of 10, 30, 90%. Each filter is made from two fused silica plates assembled by optical contact. The 90% filter is made by assembling 2 clear plates. The 10% and 30% filters are made by assembling a fused silica plate which carries a film of semitransparent chromium to produce the desired transmittance, and a clear plate. Each filter assembly is placed in a metal holder provided with shutters.
2. Liquids and Cuvettes		
931b Liquid Absorbance Standards for Ultraviolet and Visible Spectrophotometry	Liquid filters made from a solution of Co and Ni metals dissolved in a mixture of nitric-perchloric acids. The pH of the solution is about 1. Prepared in the NBS Center for Analytical Chemistry.	Three solutions and a blank are delivered in sealed ampoules and have nominal absorbances of 0.1 to 0.9.
935 Crystalline Potassium Dichromate for Use as an Ultraviolet Absorbance Standard	Crystalline potassium dichromate of established purity obtained from the J. T. Baker Chemical Co., Phillipsburg, New Jersey.	The crystalline material of reagent grade purity is offered in glass bottles provided with plastic screw caps.

Certification	Use	Remarks
The transmittance of the filter is certified as for SRM 930 but only for λ 465 nm and for a nominal transmittance of 30%.	This SRM is intended as a reference source for one-point verification of the transmittance or absorbance scales of spectrophotometers.	Same as for SRM 930. SRM 2030 is particularly useful in connection with the chemical analysis of spices. A copy of the Certificate is included in the Appendix.
The transmittance of each filter is measured with the high-accuracy spectrophometer at λ 250, 280, 340, 360, 400, 465, 500, 546.1, 590, 635 nm.	This SRM is intended as a reference source for the verification of the transmittance and absorbance scales of conventional spectrophotometers in the ultraviolet and visible spectral region.	SRM 2031 has a good optical neutrality and can be used to verify spectrophotometers with wide spectral bandpasses from about 200 to 800 nm (with possible extension to λ 3 μm). For further details consult the Certificate in the Appendix; see, also NBS Special Publication 260-68.
The absorbance of each solution was measured at λ 302, 395, 512, 678 nm using spectral bandpasses of 1.0, 1.7, 2.0, 6.5 nm respectively. The certification is made with an uncertainty of about ±1.0%.	This SRM was primarily intended for the verification of the accuracy of transmittance or absorbance scales of spectrophotometers.	Requires the use of SRM 932 and of narrow spectral bandpasses. Provides only one certified value in the ultraviolet. For further details consult the Certificate included in the Appendix.
The apparent specific absorbance is certified for five concentrations at λ 235, 257, 313, 345, 350 nm using spectral bandpasses of 1.2, 0.8, 0.8, 0.8, 0.8 nm respectively, using SRM 932.	This SRM is intended to be used as a reference standard for the verification of the accuracy and linearity of the absorbance scale of spectrophotometers.	Requires accurate preparation of solutions of potassium dichromate in 0.001N perchloric acid. Requires the use of SRM 932 and of narrow spectral bandpasses. Can be used only in the ultraviolet. For further details consult the Certificate included in the Appendix; see also NBS Special Publication 260-54.

Table 2. F. Spectrophotometry and Fluorescence - continued.

SRM	Material	Composition
932 Quartz Cuvette for Spectrophotometry	All-quartz cuvette made of non-fluorescent fused silica of optical quality. The first 100 cuvettes were prepared by the NBS Optical Shop; subsequent units were prepared to NBS specifications by Starna, Ltd., Dayenham, England.	Each cuvette is made entirely of fused silica and is provided with a Teflon stopcock. The transparent windows are attached to the body of the cuvette by direct fusion. The cuvettes are stress-relieved by proper annealing. The cuvette is stored in a transparent plastic container.
3. Stray Light		
2032 Potassium Iodide for use as a Stray Light Standard	Crystalline potassium iodide of established purity, obtained from the J. T. Baker Chemical Co., Phillipsburg, New Jersey.	The reagent-grade, crystalline KI is 99.8% pure as indicated by the manufacturer. Moisture is 0.007%. Homogeneity was tested by absorbance measurements at 265, 267, and 270 nm, and was found adequate.
2033 Potassium Iodide for use as a Stray Light Standard with Radiation Attenuator	Crystalline potassium iodide of established purity, and two semi-transparent evaporated metal-on-fused silica filters contained in a metal holder provided with shutters. The KI was obtained from the J. T. Baker Chemical Co., Phillipsburg, New Jersey. The filters and the holder were made by the NBS Instrument Shop.	Potassium iodide as for SRM 2032. Two semi-transparent evaporated chromium-on-fused silica (non-fluorescent) filters of optical quality.
4. Wavelength		
2009 Didymium Glass Filter for Checking the Wavelength Scale of Spectrophotometers	The didymium glass was prepared by Corning Glass Works, Corning, New York, as Corning 5120 Filter Glass.	Rare earth oxides in a glass matrix. This filter is 1 cm wide, 3 cm high, and 3 mm thick. Each filter is placed in a metal holder which fits into the cuvette holder of the spectrophotometer.
2010 Didymium Glass Filter for Checking the Wavelength Scale of Spectrophotometers	Same as SRM 2009.	Rare earth oxide in a glass matrix. This filter is 5.1 cm square and 3 mm thick.

Certification	Use	Remarks
The nominal inner pathlength is 10 mm and it is certified at 10 points along the height of the transparent windows with an uncertainty of ±0.0005 mm. The cuvettes are 12.5 mm square and 48 mm high.	To be used when accurate transmittance or absorbance measurements are made on liquid samples. Should be used in conjunction with SRM's 931, 935, 2032, and 2033.	For further details consult the Certificate included in the Appendix; see also NBS Special Publication 260-32.
The specific absorbances were measured with the high-accuracy spectrophotometer at 240, 245, 250, 255, 265, 270, and 275 nm and 23.5 °C using a spectral bandpass of 0.2 nm. The measurements were performed using SRM 932 quartz cuvettes.	SRM 2032 is intended to be used to assess heterochromatic stray light in the ultraviolet region below 260 nm, in absorption spectrophotometers. It is recommended that SRM 932 be used in the measurements.	SRM 2032 should be stored in the original, low-actinic glass bottle and cardboard container, protected from exposure to light and humidity. The estimated stability is 3 years.
Same as for SRM 2032. The transmittance of the evaporated metal-on-fused silica filters was measured at 255 nm with the high-accuracy spectrophotometer. The nominal value is 10% for each filter, and a combined value of 1%.	SRM 2033 is intended to be used to assess heterochromatic and isochromatic stray light in absorption spectrophotometers.	Same as for SRM 2032. The two semi-transparent evaporated metal-on-fused silica filters in the metal holder should be stored in the plastic container provided with SRM 2033.
The wavelengths of maximum absorption was determined with a high-precision spectrophotometer for bandwidths in the range 1.5 to 10.5 nm and for 14 to 24 wavelengths in the range 400 to 760 nm. The instrument has a wavelength accuracy of 0.04 nm.	The filters are intended to be used in calibrating the wavelength scale in the visible spectral region for spectrophotometers having nominal bandwidths in the range 1.5 to 10.5 nm.	SRM 2009 was not measured individually. It is recommended for most applications. For further details consult NBS Special Publication 260-66.
Same as SRM 2009.	Same as SRM 2009.	Same as SRM 2009.

Table 2. F. Spectrophotometry and Fluorescence - continued.

SRM	Material	Composition
2013 Didymium Glass Filter for Checking the Wavelength Scale of Spectrophotometers	Same as SRM 2009.	Same as SRM 2009.
2014 Didymium Glass Filter for Checking the Wavelength Scale of Spectrophotometers	Same as SRM 2009.	Same as SRM 2009.
2034 Holmium Oxide for use as Wavelength Standard in Spectrophotometry and Fluorescence Spectrometry. (Planned SRM)	Holmium oxide of established purity dissolved in perchloric acid.	Holmium oxide is to be offered in solutions in sealed 10 mm quartz cuvettes.
5. Fluorescence		
936 Quinine Sulfate Dihydrate	The quinine sulfate dihydrate was a special lot of material obtained from J. T. Baker Chemical Co., Phillipsburg, New Jersey.	SRM 936 contains 1.7% impurities determined by liquid chromatography and believed to be dihydroquinine sulfate dihydrate. The water content is 4.74±0.05% by the Karl Fisher method and 4.57±0.04% weight loss. Theoretical value is 4.60%.

Certification	Use	Remarks
Every filter was measured individually, hence the data provide a more accurate representation of the optical properties of the individual filter.	Same as SRM 2009.	SRM 2013 was measured individually and should be used when assurance in the evaluation of an instrument's wavelength scale is highly critical. For further details consult NBS Special Publication 260-66.
Same as SRM 2013.	Same as SRM 2009.	Same as SRM 2013.
The wavelengths of maximum absorption of holmium oxide solutions in perchloric acid will be determined for various spectral bandpasses at the nominal wavelengths; 279, 288, 338, 361, 386, 418, 453, 536, 637 nm.	This SRM is intended to be used as a reference standard for the verification of the accuracy of the wavelength scale of absorption and fluorescence spectrometers, in the ultraviolet and visible spectral regions.	Holmium oxide solutions in perchloric acid were selected as a wavelength standard because the absorption bands are narrower than those of the holmium oxide glass. The use of solutions in conjunction with a diffusing screen (Teflon) placed at 45° in the 10 mm cuvette permits the use of SRM 2034 for the wavelength calibration of fluorescence spectrometers. The issuance of SRM 2034 is projected for the end of 1981.
The material is certified for the relative molecular emission spectrum $E(\lambda)$, in radiometric units for a solution of 1.28×10^{-6} mol/L in 0.105 mol/L perchloric acid, using an excitation wavelength of 347.5 nm. The certified values of the molecular emission spectrum at 5 nm intervals from 375 to 675 nm are given. This certification was made with the NBS reference fluorescence spectrometer.	For evaluation of methods and the calibration of fluorescence spectrometers. A solution of 0.1 mg/mL in 1000 mL 0.105 mol/L perchloric acid is recommended. It should be stored in the dark in a well-stoppered glass bottle. This solution is stable for three months. SRM 936 is for "in vitro" diagnostic use only.	The material should be kept in its original bottle and stored in the dark at 30 °C or less. Under these conditions SRM 936 is stable for three years. (See NBS Special Publication 260-64.)

Table 3. NBS Publications in the "260 Series" Related to Clinical Chemistry.

Publication	Short Title	SRM No.
260-32	Quartz Cuvettes for Spectrophotometry	932
260-36	Calcium in Serum (Method)	915
260-48	Clinical Thermometers	933, 934
260-51	Glass Filter for Spectrophotometry	930
260-53	pH Measurements	922, 923 (and 15 others)
260-54	Potassium Dichromate UV Standard	935
260-60	Sodium in Serum (Method)	919
260-63	Potassium in Serum (Method)	918
260-64	Quinine Sulfate Fluorescence Standard	936
260-66	Didymium Glass Wavelength Standards	2009, 2010, 2013, 2014
260-67	Chloride in Serum (Method)	918, 919
260-68	Metal-on-Quartz Filters	2031
260-69	Lithium in Serum (Method)	924
260-xx	Potassium Iodide Stray Light Standard	2032, 2033
260-72	Antiepilepsy Drug Level Assay Standard	900

While all of the publications listed in Table 3 should prove valuable to users of SRM's, two provide additional information that should benefit a much wider audience. In SP 260-51, Appendix III is a partial reprint of NBS Letter Circular 1017, Standard for Checking the Calibration of Spectrophotometers, which was first issued in 1955 and reissued in 1967, but has been out of print for several years. In SP 260-54, Appendix III is SP 260-32, Standard Quartz Cuvettes for High Accuracy Spectrophotometry; Appendix V is NBS Circular 602, Testing of Glass Volumetric Apparatus (1959); and Appendix VI is NBSIR 74-461, The Calibration of Small Volumetric Laboratory Glassware (1974).

Other NBS publications, not in the "260 Series," and a number of NBS staff authored papers have been published that deal with specific SRM's or measurement techniques used in clinical chemistry. Some of these are: SP 481, Gallium Melting Point Standard, which describes SRM 1968; SP 148, The Role of Standard Reference Materials in Measurement Systems, which describes the SRM program in general, and briefly the use of SRM's in clinical measurements; and both SP 378, Accuracy in Spectrophotometry and Luminescence Measurements, and SP 466, Standardization in Spectrophotometry and Luminescence Measurements, contain papers of particular interest to clinical chemists. Another publication that should be of interest is SP 492, Procedures Used at the National Bureau of Standards to Determine Selected Trace Elements in Biological and Botanical Materials.

Appendix I. Alphabetical Index of SRM's

CALORIMETRIC
- Combustion calorimetric
- Heat source

CANE SUGAR (Sucrose)

CARBIDES
- Tungsten carbide

CARBON
- Channel black (rubber compounding)
- Conducting black (rubber compounding)
- Gas furnace black (rubber compounding)
- Oil furnace black (rubber compounding)
- Carbon dioxide in nitrogen
- Carbon monoxide in nitrogen

CARBON STEELS

CARBON TETRACHLORIDE ON CHARCOAL

CAST IRONS
- Blast furnace
- Car wheel
- Chip form
- Ductile
- Nodular
- Solid form
- White

CAST STEEL

CELLULAR PLASTICS

CEMENTS
- Portland (chemical composition)
- Turbidimetric and fineness

CERAMIC MATERIALS
- Carbides
- Glasses
- Minerals
- Refractories

CERIUM
- Radioactivity

CESIUM
- Radioactivity

CHEMICAL STANDARDS
- Primary
  - Acid potassium phthalate
  - Arsenic trioxide
  - Benzoic acid
  - Boric acid
  - Potassium chloride
  - Potassium dichromate
  - Sodium oxalate
  - Sucrose
  - Tris(hydroxymethyl)aminomethane
  - Uranium oxide
- Intermediate purity
  - Selenium
  - Zinc

CHLORINE
- Isotopic reference
- Radioactivity

CHLOROFORM ON CHARCOAL

CHROMIUM
- Ferro (steelmaking)
- Metallo-organic compound
- Isotopic reference
- Radioactivity
- Refractories

CLAYS
- Flint
- Plastic

CLINICAL LABORATORY STANDARDS
- Antiepilepsy drug level assay
- Bilirubin
- Bovine serum albumin (powder)
- Bovine serum albumin (solution)
- Calcium carbonate
- Cholesterol
- Creatinine
- Cortisol
- Gallium melting point
- Glass filters for spectrophotometry
- D-Glucose
- Iron metal
- Lead nitrate
- Liquid filters for spectrophotometry
- Lithium carbonate
- Magnesium gluconate
- D-Mannitol
- Metals on quartz filters
- Potassium chloride
- Potassium dichromate
- Quartz cuvette for spectrophotometry
- Quinine sulfate dihydrate
- Sodium chloride
- Thermometers, enzymology
- Tris(hydroxymethyl)aminomethane
- Tris(hydroxymethyl)aminomethane HCl
- Urea
- Uric acid
- VMA

COAL
- Fly ash, trace elements in
- Mercury in
- Sulfur in
- Trace elements in

COATING THICKNESS
- Copper on steel
- Gold on copper
- Gold on copper-clad glass-epoxy laminate
- Gold on Fe-Ni-Co glass sealing alloy
- Gold on nickel
- Nickel on brass
- Nickel on steel
- Tin on steel

COBALT
- Metallo-organic compound
- Radioactivity
- See, Alloys

COLOR STANDARDS

COPPER
- "Benchmarks" (chips)
- "Benchmarks" (solids)
- Beryllium copper alloys
- Brass
  - Aluminum
  - Cartridge
  - Free cutting
  - Naval
  - Red
  - Sheet
- Bronze
  - Commercial
  - Leaded tin
  - Silicon
- Coating thickness
- Concentrate
- Cupro-nickel (chips)
- Cupro-nickel (solids)
- Freezing point
- Gilding metal
- Isotopic reference
- Linear thermal expansion
- Metallo-organic compound
- Microprobe (gold-copper wires)
- Mill heads
- Mill tails
- Nickel silver alloy

DENSITY STANDARDS
- Neutron density wire
- Photographic
- Smoke density, flaming
- Smoke density, non-flaming
- 2,2,4-trimethylpentane
- X-ray

DEXTROSE (Glucose)

1,2-DICHLOROETHANE ON CHARCOAL

DIELECTRIC
- Cyclohexane
- 1,2-dichloroethane
- Nitrobenzene

DIFFERENTIAL THERMAL ANALYSIS

p-DIOXANE ON CHARCOAL

ENVIRONMENTAL STANDARDS
- Gases
- Liquids
- Radioactivity
- Solids

ELASTICITY

ELECTRICAL RESISTIVITY

EMITTANCE

FELDSPARS
- Soda
- Potash

FERRO- (STEELMAKING ALLOYS)
- Chromium
- Niobium
- Phosphorus
- Silicon

FERTILIZER
- Ammonium dihydrogen phosphate
- Phosphate rock
- Potassium dihydrogen phosphate
- Potassium nitrate

FILTERS
- Glass for spectrophotometry
- Liquid for spectrophotometry

FISSION TRACK GLASSES

FLAMMABILITY STANDARDS
- Flame spread index (hardboard sheet)
- Smoke density, flaming
- Smoke density, non-flaming
- See also, Special Reference Materials

FLUORINE IN URINE

FLUORSPAR
- Assay
- Geological

FORENSIC STANDARDS
- Glass, refractive index
- Silicone liquids, refractive index

FREEZE DRIED URINE
- Fluorine
- Mercury

FREEZING POINT STANDARDS
- Defining fixed points
  - Tin
  - Zinc
- Determined reference points
  - Aluminum
  - Copper
  - Lead
  - Mercury
  - Tin
  - Zinc

FUELS (see, environmental)
- Isooctane
- n-Heptane

GALLIUM
- Melting point

GAS TRANSMISSION
- Polyester plastic film for oxygen

GASES, ANALYZED
- Carbon dioxide in nitrogen
- Carbon monoxide in air
- Carbon monoxide in nitrogen
- Methane in air
- Methane-propane in air
- Nitric oxide in nitrogen
- Nitrogen dioxide permeation device
- Oxygen in nitrogen
- Propane in air
- Sulfur dioxide in nitrogen
- Sulfur dioxide permeation tubes

GASES IN METALS

"GASOLINE"
- Lead in
- Fuel ratings

GEOLOGICAL
- Fluorspar

GLASS
- Chemical composition
- Filters for spectrophotometry
- Fission track
- Physical properties
- Refractive index
- Spheres for sieve calibration (sizing standards)
- Stress-optical coefficient
- Trace elements in

GLUCOSE (Dextrose)
- Clinical
- Primary chemical

GOLD
- Coating thickness
  - on epoxy
  - on Fe-Ni-Co glass sealing alloy
  - on nickel
- High-purity
- Microprobe
- Vapor pressure

HEAT STANDARDS
- Calorimetric
- Freezing points
- Melting points
- Superconductive fixed points
- Thermal conductivity
- Thermal expansion
- Thermocouple materials
- Vapor pressure

HEAT SOURCE
- Zirconium-bariumchromate

HIGH-PURITY METALS
- Gold, wire and rod
- Platinum, wire
- Zinc

HIGH TEMPERATURE ALLOYS

HYDROCARBON BLENDS

HYDROGEN
- in steel (GM-1, GM-2)
- in titanium
- Radioactivity

INDUSTRIAL HYGIENE
- Benzene
- Beryllium/filter
- Carbon tetrachloride
- Chloroform
- 1,2-Dichloroethane
- p-Dioxane
- Fluorine in urine
- Mercury in urine
- Metals/filter

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 900

Antiepilepsy Drug Level Assay Standard

This Standard Reference Material (SRM) is certified for concentrations of four antiepilepsy drugs--phenytoin, ethosuximide, phenobarbital, and primidone--in a processed human serum base. It is intended for use (1) in the calibration and standardization of procedures employed in clinical laboratories for the determination of these drugs in serum, and (2) for the critical evaluation of working or secondary reference solutions prepared either in-house or supplied commercially. The certified concentrations apply to the four materials supplied, after each serum is reconstituted, following the procedures described in "Instructions for Use."

Concentration Level, μg/mL

Drug	Toxic	Therapeutic	Subtherapeutic	Blank
Phenytoin	60.7 ± 0.9	16.7 ± 0.3	4.2 ± 0.1	0
Ethosuximide	174.7 ± 0.6	75.9 ± 0.5	11.8 ± 0.4	0
Phenobarbital	103.6 ± 0.3	21.6 ± 0.2	5.3 ± 0.2	0
Primidone	18.6 ± 0.7	8.1 ± 0.2	3.6 ± 0.1	0

The uncertainties represent one standard error for the above certified values. The imprecisions observed both within and between the liquid and gas chromatographic analyses used for this certificate are included in the standard errors. The statistical analyses were made by R. C. Paule and J. Mandel.

The modified human serum base was processed, vialed and packaged by Microbiological Associates, Walkersville, Maryland. Analyses leading to certification were performed in the NBS Center for Analytical Chemistry by R. Angeles, R. G. Christensen, A. Cohen, B. Coxon, D. Enagonio, D. J. Reeder, and L. T. Sniegoski. Valuable correlating analyses were also performed by: E. Berman, Cook County Hospital, Chicago, Illinois; K. Dudley, The University of North Carolina at Chapel Hill; and H. J. Kupferberg, National Institute of Neurological and Communicative Disorders and Stroke, NIH.

The overall direction and technical measurements leading to the certification were under the chairmenship of D. J. Reeder and R. Schaffer.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Keith Kirby.

Washington, D.C. 20234
April 5, 1979
(Editorial Revision of Certificate
dated 11-17-78)

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

The analytical techniques used in the certification of this Standard Reference Material were liquid chromatography, calibrated with external standards, and gas chromatography, calibrated by bracketing and internal standards. The drug concentrations have also been determined with commercially available reagents that are based on immunoenzymatic assay principles. The mean values obtained by this methodology were close to the certified values; however, the precision was not sufficient to be included in the certification data.

The Standard Reference Material is intended for "in vitro" diagnostic use only.

WARNING: HANDLE AS IF CAPABLE OF TRANSMITTING HEPATITIS. Source material from which this serum base was derived was found non-reactive for Hepatitis B antigen when tested with licensed third-generation reagents. No known test method can provide complete assurance that products derived from human blood will not transmit hepatitis.

Instructions for Use

This Standard Reference Material is supplied as a set of four different freeze-dried preparations. They should be stored at refrigerator temperature (~ 4 °C) and should not be exposed to sunlight or ultraviolet radiation. Under such storage, the SRM is expected to be stable for at least 2 years. Samples of this SRM will be monitored. Should statistical evidence indicate a degradation of the certified properties, purchasers will be notified by NBS. It is recommended that the material not be used after 2 years from the date of purchase.

For use, it is necessary to reconstitute the freeze-dried materials with high-purity water.* When a vial is opened, remove the rubber stopper carefully so as not to dislodge any serum particles that may adhere to the stopper. Add 5.0 mL of water to the vial from a calibrated volumetric pipet or other dispenser of known accuracy. Replace the stopper and allow the contents to stand at room temperature for 20-30 min. Finally, mix the contents by gentle swirling. DO NOT shake vigorously because denaturation and frothing may result.

After reconstitution, the contents should be used within one day; otherwise, the certified values cannot be assured. Storage of the reconstituted material beyond 12-24 hours may result in deterioriation of the drugs or in degradation of the serum base.

This Standard Reference Material has been measured and certified at the Laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D.C. 20234

The date of issuance and certification of this Standard Reference Material is November 17, 1978.

*Equivalent to Type II reagent grade water as specified by the College of American Pathologists (CAP).

U. S. Department of Commerce
Philip M. Klutznick
Secretary

National Bureau of Standards
Ernest Ambler, Director

SEE ADDENDUM

National Bureau of Standards

Certificate

Standard Reference Material 909

Human Serum

This Standard Reference Material (SRM) is intended for use in evaluating the accuracy of clinical procedures for the determination of specified constituents in serum, in calibrating instruments and equipment used in these procedures, and in validating working or secondary reference materials.

CERTIFIED VALUES OF ANALYTES: The concentrations of the analytes were determined by methods having the highest accuracy, i.e., definitive methods. The certified values are given in two ways, depending on whether reconstitution is done with or without weighing the freeze-dried serum contents of a vial. Concentration values having smaller uncertainties may be obtained by weighing the freeze-dried serum and multiplying this mass by the certified concentration values of the analytes per unit mass of dried serum. These certified values appear in Table 1. When the contents of a vial are not weighed, the mean values for the certified concentrations and their uncertainties should be used. These values, which apply to all vials of reconstituted SRM 909, appear in Table 2.

NOTICE AND WARNINGS TO USERS

USE: HANDLE AS IF CAPABLE OF TRANSMITTING HEPATITIS! Although this product was tested with licensed third generation reagents and found nonreactive for the presence of hepatitis B surface antigen (HB_SAG), no known test method can offer assurances that products derived from human blood will not transmit hepatitis.

SRM 909 IS INTENDED FOR "IN VITRO" DIAGNOSTIC USE ONLY.

STORAGE: The freeze-dried serum should be stored in a refrigerator at a temperature between 2 and 8 °C. It should not be frozen nor exposed to sunlight or ultraviolet radiation. Under the recommended storage conditions, this SRM is expected to be stable for at least one year; should evidence indicate a more rapid degradation of the certified properties, purchasers will be notified by NBS. The material is not certified for use after one year from date of purchase.

The statistical analysis of the data was performed by K.R. Eberhardt of the Statistical Engineering Division.

The overall direction and technical measurements leading to the certification were under the chairmenship of E. Garner, H.S. Hertz, T.J. Murphy, D.J. Reeder, R. Schaffer and E. White V.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, D.C. 20234
September 15, 1980
January 13, 1981 (revision)

George A. Uriano, Chief
Office of Standard Reference Materials

(over)

Instructions for Use

HANDLE AS IF CAPABLE OF TRANSMITTING HEPATITIS! SRM 909 is supplied as a set of six vials of freeze-dried human serum and six vials of high-purity, diluent water for use in reconstituting the serum.

Two procedures for reconstituting SRM 909 are described. Selection of a procedure depends on the uncertainties required for the concentrations of the analytes. If lower uncertainties than those shown in Table 2 are required, the freeze-dried serum contents of a vial must be weighed as described in Procedure A.

Procedure A. Reconstitution with weighing of the freeze-dried serum: Completely remove label and adhesive by scraping the vial and then wiping it with a tissue moistened with a solvent, such as acetone or ethanol. Scratch an identification on vial. Remove metal closure and lightly tap bottom of vial to dislodge any serum particles adhering to the stopper. Dislodge stopper to equalize air pressure, then replace, wipe surface of vial, and weigh to the nearest 0.1 mg. (Use a clean empty vial of the same size as a tare.) Carefully remove stopper to avoid possible loss of serum particles. Use a Type 1 Class A volumetric transfer pipet or other dispenser of known accuracy to slowly add 10.00 ± 0.02 mL of the diluent water at 20 - 25 °C to the sides of the vial while continually turning the vial. Replace stopper, swirl vial two or three times, and let stand for 10 minutes. Mix contents by gently swirling, let stand for approximately 30 minutes, swirl again, let stand 10 minutes, and finally invert the vial several times. *Do NOT shake vigorously* because this will cause frothing. Total time for reconstitution is approximately 1 hour. After reconstitution, use contents as soon as possible. If not used immediately, store between 2 and 8 °C until ready for use, preferably within 8 hours. After the reconstituted serum has been used, clean and dry the vial and its stopper. Reweigh after replacing stopper. The tare is reweighed at the same time to compensate for changes in temperature and humidity. The mass of dry serum is given by the difference between the original and final weighings.

The concentration of an analyte, after the contents of a vial is weighed and reconstituted with 10.00 mL of diluent water, is calculated by multiplying the mass of freeze-dried serum, in grams, by the certified concentration of the analyte per gram of freeze-dried serum given in Table 1. For example, if the mass of freeze-dried serum in a vial is 0.8703 g, the concentration of uric acid in this vial would be:

$$0.5681 \text{ mmol}\cdot\text{L}^{-1}\cdot\text{g}^{-1} \times 0.8703 \text{ g} = 0.4944 \text{ mmol/L}.$$

The uncertainty is also calculated similarly and for this example would be:

$$0.0050 \text{ mmol}\cdot\text{L}^{-1}\cdot\text{g}^{-1} \times 0.8703 \text{ g} = 0.0044 \text{ mmol/L}.$$

Table 1. Certified Analyte Concentrations and Uncertainties per Gram of Freeze-Dried Serum after Reconstitution of SRM 909 according to Procedure A.

Analyte[1]	Concentration,[2] per gram, $\text{mmol}\cdot\text{L}^{-1}\cdot\text{g}^{-1}$	Uncertainty,[3] per gram, $\text{mmol}\cdot\text{L}^{-1}\cdot\text{g}^{-1}$
Calcium[a]	3.560	± 0.013
Chloride[a]	128.0	± 1.5
Glucose[a]	7.811	± 0.095
Lithium[a]	1.945	± 0.033
Potassium[a]	4.1546	± 0.0098
Uric Acid[a]	0.5681	± 0.0050

1. Analytical Methods
   a. Isotope dilution mass spectrometry.
2. The certified concentrations apply to reconstituted serum at room temperature (20-25 °C).
3. The uncertainties represent 95%/99% statistical tolerance limits for the concentrations per gram of dry serum reconstituted with 10.00 mL of high-purity water. The tolerance limits are constructed so that, at a confidence level of 95%, they will contain the concentration/mass ratios for at least 99% of the vials.

Procedure B. Reconstitution of SRM 909 without weighing freeze-dried serum: Remove metal closure and lightly tap bottom of vial to dislodge any serum particles on stopper. Carefully remove stopper to avoid possible loss of serum particles. As described in Procedure A, reconstitute with 10.00 ± 0.02 mL of the diluent water, and use immediately or store between 2 and 8 °C until ready for use, preferably within 8 hours. Table 2 gives the certified concentrations of the constitutents and the tolerance limits for use with this procedure.

Table 2. Certified Concentrations and Uncertainties for Analytes in Reconstituted SRM 909 for Use with Procedure B.

Analyte[1]	Concentration[2], $mmol \cdot L^{-1}$	Uncertainty[3], $mmol \cdot L^{-1}$
Calcium[a]	3.02	+0.17 −0.06
Chloride[a]	108	+7 −3
Glucose[a]	6.62	+0.44 −0.20
Lithium[a]	1.65	+0.12 −0.06
Potassium[a]	3.52	+0.19 −0.06
Uric Acid[a]	0.481	+0.031 −0.012

1. Analytical Methods:
   a. Isotope dilution mass spectrometry.
2. The certified concentrations apply to reconstituted serum at room temperature (20-25 °C).
3. The uncertainties represent 95%/95% statistical tolerance limits for the concentrations, and reflect the combined effects of measurement imprecision and the variability of the mass of dry serum among vials. They are constructed so that, at a confidence level of 95%, they will include the concentrations of at least 95% of all vials of SRM 909, when reconstituted according to Procedure B.

The major source of uncertainty in the certified concentrations shown in Table 2 is caused by vial-to-vial variability in the mass of freeze-dried serum. Information on the relation between mass variability and concentration was obtained directly during the course of experimentation by weighing the contents of each vial as an intermediate step in determining the concentration of an analyte. Supplementary information on the variability in mass was obtained from a differential refractometry experiment in which over 170 vials were analyzed. The data obtained from these two sources indicate that the distribution of masses is non-Gaussian and skewed in the direction of high mass. The largest two masses encountered were 5.2% and 2.7% above the mean mass, 0.8469 g. All other masses were contained in an interval from 1.5% below to 1.8% above the mean.

Source of Material: The human serum for SRM 909 was processed, vialed, and packaged by Hyland Division, Travenol Laboratories Inc., Round Lake, Illinois.

Analyses were performed in the NBS Center for Analytical Chemistry by I.L. Barnes, K.A. Brletic, R.G. Christensen, A. Cohen, J.W. Gramlich, W.R. Kelly, L.R. Machlan, J.R. Moody, L.J. Powell, L.T. Sniegoski, and M.J. Welch (Research Associate, College of American Pathologists).

This Standard Reference Material has been measured and certified at the Laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standards Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D.C. 20234

The date of issuance and certification of this Standard Reference Material is September 15, 1980.

U. S. Department of Commerce
Philip M. Klutznick
Secretary

National Bureau of Standards
Ernest Ambler, Director

Addendum to

National Bureau of Standards Certificate of Analysis

Standard Reference Material 909

Human Serum

ADDITIONAL CERTIFICATION

The following certified values are to be added to Tables 1 and 2.

Table 1. Certified Analyte Concentrations and Uncertainties per Gram of Freeze-Dried Serum after Reconstitution of SRM 909 according to Procedure A.

Analyte[1]	Concentration,[2] per gram, $mmol \cdot L^{-1} \cdot g^{-1}$	Uncertainty,[3] per gram, $mmol \cdot L^{-1} \cdot g^{-1}$
Cholesterol[a]	4.346	±0.030
Magnesium[a]	1.425	±0.072

Table 2. Certified Concentrations and Uncertainties for Analytes in Reconstituted SRM 909 for Use with Procedure B.

Analyte[1]	Concentration[2], $mmol \cdot L^{-1}$	Uncertainty[3], $mmol \cdot L^{-1}$
Cholesterol[a]	3.68	+0.22 −0.08
Magnesium[a]	1.21	+0.14 −0.10

Washington, D.C. 20234
November 14, 1980

George A. Uriano, Chief
Office of Standard Reference Materials

U. S. Department of Commerce
Malcolm Baldrige
Secretary
National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 910
Sodium Pyruvate

This Standard Reference Material (SRM) is certified as a chemical of known purity. It is intended primarily for use in the calibration and standardization of procedures for pyruvate, lactic dehydrogenase, and glutamic-pyruvic transaminase determinations in clinical analyses, and for critical evaluation of the routine working or secondary reference materials used in these procedures.

Sodium Pyruvate[1]	98.7	± 0.2	weight percent
Sodium 2-hydroxy-4-keto-2-methylpentanedioate[1] (parapyruvate)	0.9	± 0.2	weight percent
Moisture[2]	(0.28)		weight percent
Methanol	(0.21)		weight percent
Water-insoluble matter	(0.004)		weight percent

[1]The certified pyruvate and parapyruvate concentrations were obtained by taking weighted averages for results from two separate methods of analysis. Measurements were made at NBS using high-performance liquid chromatography (HPLC) and high-field, proton NMR spectroscopy at 400 MHz. For each certified average, the relative weights were calculated based on the internal (within-method) and the between-method variance components. The standard errors of the certified concentrations were also calculated using combinations of both the variance components. The individual results for each method of analysis were: NMR - pyruvate, 98.83 ± 0.05; parapyruvate, 0.68 ± 0.05; HPLC - pyruvate, 98.50 ± 0.03; parapyruvate, 1.02 ± 0.03 weight percent.

[2]Values in parentheses are not certified because they were determined by one method only.

All reported uncertainties are stated as plus or minus one standard error of the listed value.

NOTICE AND WARNINGS TO USERS

This Standard Reference Material is intended for "in vitro" diagnostic use only.

Storage:

SRM 910 should be stored in the tightly capped bottle at 2-6 °C. It should be allowed to warm to room temperature before opening. Under proper storage, this material should be stable for at least 5 years. If the purity of the material degrades beyond the limits certified, purchasers will be notified by NBS. This material is not certified for use after 5 years from date of purchase.

The sodium pyruvate used for this SRM was obtained from the Sigma Chemical Company, St. Louis, Missouri. Analyses and physical determinations were performed at NBS in the Organic Analytical Research Division by B. Coxon, A. Cummings, J. Lee, S. Margolis, and L. Sniegoski.

The statistical analysis of the data was made by R. Paule, NBS National Measurement Laboratory.

The overall direction and coordination of the technical measurements leading to the certification were under the chairmanship of S. Margolis and B. Coxon.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, D.C. 20234
May 15, 1981

George A. Uriano, Chief
Office of Standard Reference Materials

(over)

Liquid chromatography of 3-mg samples of this SRM, dissolved in water, was performed by using a 150 x 0.9 cm column packed with Sephadex G-15, operated at a pressure of 500 kPa. The eluant was 0.03 mol/L sodium phosphate buffer at pH 8.0. Elution was monitored by measurement of refractive index and transmittance at 254 nm. A major peak and two minor peaks with retention times of 0.65 and 0.80 relative to the major peak were detected. The material corresponding to each peak was collected, lyophilized, checked for chromatographic purity by chromatography on a Sephadex G-15 column, and characterized by both proton and carbon-13 (^{13}C) NMR spectroscopy. Chromatography on Sephadex G-15 of chromatographic fractions before and after lyophilization showed that no changes occurred on lyophilization of the separated materials nor did new products form during the process of chromatography.

The major peak exhibited proton and ^{13}C NMR spectra that are characteristic of sodium pyruvate: at pH 6 and pH 8, proton NMR indicated that approximately 7.5 percent and 4.7 percent, respectively, of the pyruvate is hydrated at the C-2 position. The impurity at the relative retention time 0.80 (the larger of the minor peaks) appears to be a single compound which exhibits an NMR spectrum characteristic of the linear form of the parapyruvate. The impurity at the relative retention time of 0.65 (present at only trace levels) was located at the void volume of the column, and NMR analysis of the lyophilized material suggested that this material was a mixture of oligomers of sodium pyruvate.

The parapyruvate content was found to be 0.68 ± 0.05 percent from methyl proton NMR peak intensities and 1.02 ± 0.03 percent from chromatographic measurements. The reasons for the discrepancy in the estimates by NMR and HPLC is unclear. It does not appear to be attributable to a contaminant in that HPLC peak, to differences in the extinction coefficients of the pyruvate and parapyruvate, or to differences in the intensities of the NMR signals of the methyl group of these compounds.

From signal measurements, ^{13}C NMR spectroscopy indicated the presence of less than 0.6 mol percent of organic impurities other than parapyruvate and methanol in the SRM. Proton NMR spectroscopy at 400 MHz was performed to determine the content of parapyruvate and methanol. The spectroscopy was performed at ambient temperature using a data set of 16,384 points, a 30° pulse, and a relaxation delay of 6.26 s between pulses.

Samples for proton NMR analysis were prepared by dissolving 240 mg of the SRM in 0.5 mL of 9:1 v/v water-deuterium oxide. No significant differences in parapyruvate or methanol content were observed for solutions that were analyzed by signal averaging over periods ranging from 7 minutes to 3 hours. After drying the SRM at 93 °C and 2.5 kPa for 15 hours, the methanol content was 0.16 percent, indicating that methanol is not readily removed by such treatment.

The structure of the parapyruvate was proven by means of ^{13}C NMR spectroscopy at 22.6 MHz using a data set of 8,192 points, a 30° or 45° pulse, a relaxation delay of 5 s, and solutions prepared by dissolving 1-g portions of the SRM in 2-mL aliquots of 9:1 v/v water-deuterium oxide. ^{13}C spectral assignments were confirmed by the use of off-resonance proton decoupling techniques, proton coupled spectra, and measurements of the spin-lattice relaxation times of sodium pyruvate and parapyruvate, using the fast inversion recovery method.

The ultraviolet absorption spectrum of sodium pyruvate in distilled water at pH 5.9 exhibited an absorbance maximum at 316 nm and a minimum at 289 nm. The molar extinction coefficient at 316 nm is 18.14 ± 0.04 L mol^{-1} cm^{-1}. Parapyruvate exhibited an absorbance maximum at 326 nm and a minimum at 301 nm. The molar extinction coefficient is 25 L mol^{-1} cm^{-1}. The absorbance of the SRM above 400 nm is less than 0.3 percent of the absorbance at 316 nm.

Microchemical analysis yielded these values, in percent: carbon, 32.64 ± 0.08; hydrogen, 2.84 ± 0.02; sodium, 20.73 ± 0.03; and oxygen, 43.49 ± 0.06. Calculated percentages based on $C_3H_3NaO_3$ are 32.74, 2.74, 20.89, and 43.91, respectively.

The homogeneity of the SRM, as determined by liquid chromatography and by proton and ^{13}C NMR spectroscopy, was found to be satisfactory.

This SRM is intended for "in vitro" diagnostic use only.

This material is for use as a standard in clinical chemistry. Stock solutions should be prepared daily with distilled water. The pH of a solution of the SRM in distilled water at a concentration of 250 mg/mL is 5.90 ± 0.06. Such solutions are stable for several weeks in the frozen state at −20 °C. However, at 20 °C and at an initial pH 5.90, the concentration of parapyruvate doubles in five days, and triples within 24 hours if the pyruvate is dissolved in 0.25 mol/L potassium phosphate at pH 8.0 and 20 °C. With the same solvent adjusted to pH 9, 50 percent of the pyruvate dimerizes within 24 hours.

U. S. Department of Commerce
Philip M. Klutznick
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 911a

Cholesterol

A. Cohen, W. May, and R. Schaffer

This Standard Reference Material is certified as a chemical of known purity. It is intended primarily for use in the calibration and standardization of procedures for cholesterol determinations employed in clinical analysis and for routine critical evaluation of the daily working standards used in these procedures.

Purity . . . . 99.8 ± 0.1 percent

The selenium dioxide test for lathosterol [1] was negative.

The purity was determined by the use of liquid chromatography, gravimetric recovery, and thin-layer chromatography. The most recent monitoring of purity, completed in February 1980, was performed by the use of mass spectrometry, capillary column gas chromatography, and thin-layer chromatography. The results showed nonsignificant changes in the levels of impurities.

This Standard Reference Material should be stored in the tightly capped bottle in a desiccator at 0°C. It should be allowed to warm to room temperature before opening. If this procedure is followed, drying is unnecessary.

The cholesterol used for this Standard Reference Material was obtained from the J. T. Baker Chemical Company of Phillipsburg, N.J. Analyses and physical determinations were performed by A. Cohen, S. P. Cram, E. R. Deardorff, D. H. Freeman, H. S. Hertz, W. May, R. Schaffer, W. P. Schmidt, C. P. Talley, and J. K. Taylor. The additional analyses were performed by S. Chesler, B. Coxon, L. Sniegoski, and E. White V.

The overall direction and coordination of technical measurements leading to the certification were under the chairmanship of R. Schaffer.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D.C. 20234
March 5, 1980
(Revision of Certificate
dated 11-1-74)

George A. Uriano, Chief
Office of Standard Reference Materials

(over)

Liquid chromatography was performed on this material using a column packed with poly-2-methyl-5-vinyl-pyridine/divinylbenzene. The sample size was exactly 1 g cholesterol (measured to the nearest 0.1 mg). The eluant was chloroform and the progress of the elution was monitored by the measurement of transmittance of effluent at 254 nm. Two peaks due to impurities were detected at 0.7 and 0.8 retention time relative to the cholesterol peak. Recovery of the sample by gravimetry corrected for the blank due to column bleed, was 100.0_4 percent. The weighed effluent residues were examined by thin-layer chromatography on 250-μm layers of silica gel using 1:1 (v/v) ethyl acetate--heptane as developing solvents. Spraying with 1:5 (v/v) sulfuric acid--methanol followed by heating to 110 °C revealed two impurities at migration distances of 1.53 and 1.16 relative to that of cholesterol. These impurities, found in fractions representing a combined weight of 0.08 percent of the intial cholesterol, corresponded to the impurities detected by prior liquid chromatography at retention times of 0.7 and 0.8, respectively.

Gas-chromatographic examination, using Gas-Chrom Q treated with 3 percent OV-17 as the stationary phase at 240 °C, showed impurity peaks at retention times 0.5, 0.80, and 1.2 relative to cholesterol.

Non-destructive neutron activation analysis of cholesterol samples sealed in high-purity quartz ampoules prior to irradiation indicated a bromine content of 12 μg/g, a chlorine content of 0.7 μg/g, and an iodine content of less than 0.8 μg/g. The estimated accuracy of these measurements was within 20 percent.

The infrared-absorption spectrum of SRM 911a was the same as that of SRM 911. The ultraviolet-absorption spectrum of SRM 911a (4 percent solution in methylene chloride) showed very little absorption in the range 400 to 240 nm. This absorption is less than that exhibited by SRM 911 under similar conditions.

The melting point of this material, measured in a sealed tube under vacuum, is 149.0 to 149.4 °C. The specific rotation $[a]_D^{20}$ is −0.700 rad (−40.1°) (c, 1; $CHCl_3$). Microchemical analysis found carbon 83.86 ± 0.05 wt. percent, hydrogen 12.00 ± 0.05 weight percent; theoretical percentages based on $C_{27}H_{46}O$ are 83.87 and 11.99, respectively.

Heating the material at 100 °C at 3Pa (0.02 mm Hg) resulted in a nearly constant loss in weight of 0.01 percent per hour over a 1.5 day period. Such a volatilization may be related to degradation and/or sublimation processes.

Precautions

This Standard Reference Material is intended for "in vitro" diagnostic use only.

The Standard Reference Material should be stored in a tightly closed bottle kept in a refrigerator or freezer. It should not be subjected to heat, direct sunlight, or artificial sources of ultraviolet radiation. For extended periods of storage after opening, it is recommended that the material be kept at or below −15 °C in a desiccator under inert gas. Experience at NBS, where the material is stored under inert gas at −15 °C, indicates the material may be stable for 10 years. If the purity of control material at NBS degrades beyond the limits certified, purchasers will be notified by NBS. However, if the material is stored under ordinary laboratory conditions in a refrigerator, it is recommended that the material should not be used after 3 years from the date of purchase. If it is stored in the dark at room temperature, it is recommended that the material not be used after six months from the date of purchase.

This material is for use as a standard in clinical chemistry. A stock standard solution of cholesterol in ethanol (5 mmol/L) may be prepared by dissolving 194 mg of SRM 911a in 50 mL of warm absolute ethanol in a 100-mL volumetric flask, allowing the solution to cool, and diluting to exactly 100 mL with ethanol [2]. It should be stored in an all-glass, tightly stoppered bottle at 0 °C. Under such conditions this solution should be stable for about 4 months [3].

Because methods for the determination of cholesterol vary widely, users should follow the instructions included with each method for preparing the working standard solution from the stock standard solution.

Solutions of cholesterol in glacial acetic acid gradually form cholesteryl acetate when stored and therefore may cause errors in calibration [4].

All constituted solutions of cholesterol should be clear and display no turbidity.

References:

[1] Fieser, L. F. ,Cholesterol and Companions. III, Cholestanol, Lathosterol and Ketone 104, J. Am. Chem. Soc. 75, 4395 (1953).

[2] Fundamentals of Clinical Chemistry, N. Tietz, editor, p. 358, W. B. Saunders Co., Philadelphia, Pa. (1970).

[3] Henry, R. D., Clinical Chemistry, Principles and Technics, p. 854, Hoeber Medical Division, Harper & Rowe, New York, N.Y. (1967).

[4] Klein, B., and Kleinman, N.B., Esterification of Cholesterol in Glacial Acetic Acid., Clin. Chem. 20, 90 (1974).

This Standard Reference Material has been measured and certified at the laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D.C. 20234

The date of issuance and certification of this Standard Reference Material was June 6, 1974.

SRM 911a

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards
Certificate of Analysis

Standard Reference Material 912a

UREA

This Standard Reference Material is certified as a chemical of known purity for use in the calibration and standardization of procedures employed in clinical analysis.

Purity	99.9	± 0.1 percent
Moisture	0.02	± 0.003 percent
Biuret	0.02	± 0.02 percent
Ash	0.001	± 0.0007 percent
Insoluble matter	0.0001	± 0.00005 percent

The purity value has an estimated uncertainty of 0.1 percent. The errors of the moisture, biuret, ash, and insoluble matter are given as two standard errors of the mean.

The urea used for this Standard Reference Material was obtained from Fluka AG, Switzerland. Analyses were performed by R. G. Christensen, B. Coxon, A. L. Cummings, J. Lee, D. J. Reeder, F. J. Savluk, and L. T. Sniegoski, of the Organic Analytical Research Division.

The overall direction and coordination of technical measurements leading to the certification were under the chairmanship of B. Coxon and D. J. Reeder.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, D.C. 20234
November 16, 1979

George A. Uriano, Chief
Office of Standard Reference Materials

(over)

Differential scanning calorimetry [1] of the Standard Reference Material indicated a purity of 99.97 ± 0.01 mole percent (exclusive of moisture content).

The moisture content of the Standard Reference Material was determined separately by Karl Fischer titration of samples ranging from three to ten grams.

Biuret was estimated spectrophotometrically by use of a 6 mol·L^{-1} solution of urea in aqueous, alkaline nickelous sulfate measured at its absorption maximum at 461 nm [2]. This determination was calibrated against dilute solutions of crystalline biuret, the purity of which was established by high resolution, pulse-Fourier transform, carbon-13 nuclear magnetic resonance (^{13}C NMR) spectroscopy of a 1 mol·L^{-1} solution of the biuret in methyl sulfoxide-$\underline{d}_6$.

Measured in a 2-cm cell, a 50 percent (wt/vol) solution of urea in water showed strong absorption (1.87 ± 0.01) at 220 nm, a weak absorption (0.036 ± 0.001) at 280 nm, and a very weak absorption (0.006 ± 0.001) at 461 nm.

The sulfated ash content of the Standard Reference Material was determined by volatilization of 25 g samples in porcelain crucibles, followed by moistening of the residue with concentrated sulfuric acid, further volatilization, and finally, ignition at 800 °C to constant weight.

Insoluble matter in the Standard Reference Material was determined by dissolution of 25 g samples in 100 mL aliquots of water, followed by filtration of the briefly boiled solutions through tared crucibles which were then dried at 110 °C.

The homogeneity of the Standard Reference Material was verified by differential scanning calorimetry, ^{13}C NMR spectroscopy, melting point determination, and moisture determination by the Karl Fischer method. Spectroscopy by ^{13}C NMR of approximately 10 mol·L^{-1} solutions of the urea in H_2O:D_2O (9:1 v/v), performed by signal averaging of 12,000 scans at 22.6 MHz showed only a single ^{13}C resonance with a chemical shift, δ_c, of 161.6 from external tetramethylsilane, and thus indicates the absence of organic impurities by this test.

The melting range of this Standard Reference Material is 133.0 to 134.0 °C, as measured in an open capillary tube heated at 0.5 °C min^{-1}.

A 10 percent (w/v) solution of this Standard Reference Material in water, free from carbon dioxide, showed a pH of 7.1 ± 0.2 at 23 °C.

This Standard Reference Material is intended for "in vitro" diagnostic use only.

This material is intended for use as a standard in clinical chemistry. A standard solution containing 20 mg per 100 mL (0.2 mg per mL) of urea nitrogen may be prepared by weighing 0.429 g urea into a one-liter volumetric flask and making to volume with ammonia-free distilled water. A few drops of ACS Reagent-Grade chloroform is to be added as a preservative. The solution should be stored in a refrigerator [3]. The concentration of urea nitrogen in this solution is approximately that of the normal level in serum. An alternate procedure [4] recommends 0.1 g sodium azide per 100 mL of solution as a preservative.

This Standard Reference Material should be stored in a well-stoppered container at room temperature (30 °C or less). It should not be subjected to heat, moisture, or direct sunlight during storage. Refrigerated storage is recommended, but the material should be allowed to warm to room temperature before opening the container. Under proper storage, experience at NBS indicates this material to be stable for at least 5 years. If the purity of the material degrades beyond the limits certified, purchasers will be notified by NBS. It is recommended that this material not be used after 5 years from the date of purchase.

The standard urea nitrogen solution (20 mg/100 mL), prepared as described above, is stable for 3 months when refrigerated at 4 °C in a well-stoppered, all-glass container.

All constituted solutions of urea should be clear and without indications of bacterial growth of any kind.

References:

[1] Plato, C., and Glasgow, A.R., Jr., Anal. Chem. 41, 330 (1969).

[2] Reeder, D.J., and Savluk, F.J., unpublished method.

[3] Henry, R.D., Clinical Chemistry, Principles and Practice, pp. 262-276, Hoeber Medical Division, Harper & Row, New York, N.Y. 10016 (1967).

[4] Tietz, N.W., Fundamentals of Clinical Chemistry, pp. 718-722, W. B. Saunders Co., Philadelphia, Pa. 19105 (1970).

This Standard Reference Material has been measured and certified at the laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D.C. 20234

The date of issuance and certification of this Standard Reference Material was November 1979.

U. S. Department of Commerce
Frederick B. Dent
Secretary
National Bureau of Standards
Richard W. Roberts, Director

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 913
URIC ACID

This Standard Reference Material is certified as a chemical of known purity. It is intended primarily for use in the calibration and standardization of procedures for uric acid determinations employed in clinical analysis and for routine critical evaluation of daily working standards used in these procedures.

Purity	99.7 percent
Volatile matter	0.14 percent
Ash	0.057 percent

The value of the purity has an estimated inaccuracy of 0.1 percent.

The ash was found to be composed principally of salts of sodium and potassium. Aluminum, calcium, iron, phosphorus, and silicon were found in proportions estimated to be between 1 and 10 percent of the ash. Cobalt, copper, manganese, nickel, and zinc were each present to an extent not exceeding 1 percent of the ash.

The uric acid used for this Standard Reference Material was obtained from the Pfanstiehl Laboratories, Inc., of Waukegan, Illinois. Analyses were performed by D. P. Enagonio, R. A. Paulson, W. P. Schmidt, V. C. Stewart, R. S. Tipson, and B. F. West of the Analytical Chemistry Division.

The overall direction and coordination of technical measurements leading to the certification were under the chairmanship of R. Schaffer.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D. C. 20234
September 24, 1968
Revised November 23, 1973

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

The uric acid used in this Standard Reference Material was found to be homogeneous by paper and thin–layer chromatography with several solvent systems. This homogeneity was further verified by gas–liquid chromatography of the trimethylsilylated material.

Volatile material was determined by heating the sample overnight at 110 °C.

An emission spectrometric analysis for metallic constituents in the ash from this Standard Reference Material showed the following present as major constituents: sodium, potassium, aluminum, calcium, iron, phosphorus, and silicon. Neutron activation analysis of the bulk Standard Reference Material indicated the presence of the following approximate concentrations of elements: sodium, 170 ppm; copper, 2.4 ppm, manganese, 0.028 ppm.

The ultraviolet absorption spectra of the material exhibited the following absorption maxima:

In lithium carbonate solution, pH = 7.6

at 292 nm, ϵ_{max} = 12,560 ± 10

at 236 nm, ϵ_{max} = 10,000 ± 80

In a glycine-buffered solution, pH = 9.6

at 292 nm, ϵ_{max} = 12,650 ± 50

at 234 nm, ϵ_{max} = 9,890 ± 50

The measures of uncertainty given are the standard deviation of a single measurement. They should not be considered to be a certified measure of inaccuracy for the extinction coefficients.

A 100–g sample of uric acid was extracted successively with 500 cm^3 each of water, absolute ethanol, benzene, and water. The ultraviolet absorption spectrum of each of these extracts showed no absorption bands other than those of uric acid. Only uric acid was detected by thin–layer chromatography of these same extracts.

This Standard Reference Material is for "in vitro" diagnostic use only.

This material is for use as a standard in clinical chemistry. A "stock standard" solution containing 1 mg/ml of uric acid may be prepared as follows. Transfer 1.00 g of SRM 913 to a 1000-ml volumetric flask. Dissolve separately 0.60 g of lithium carbonate (SRM 924) in 150 ml warm deionized water, filter, and heat filtrate to 60 °C. A slight turbidity at this point may be ignored. Add the warm lithium carbonate solution to the uric acid (SRM 913) and mix until completely dissolved. It may be necessary to warm the flask under hot running water during mixing. Cool to room temperature. The solution usually remains slightly turbid. Add 20 ml of 40% formaldehyde. Dilute to about 500 ml with deionized water, then slowly add with mixing 25 ml of 1 N sulfuric acid. Dilute to the mark with deionized water, mix well, and place in a glass-stoppered, dark-brown bottle and store in a refrigerator at 4 °C [1,2].

A "working" standard solution containing 5 μg/ml may be prepared by transferring 0.50 ml of the above "stock" standard solution to a 100-ml volumetric flask and diluting to the mark with deionized water. Store solution in refrigerator at 4 °C. Prepare fresh weekly.

Similar standard solutions prepared without the use of formaldehyde and sulfuric acid are described by Faulkner and King [3].

This Standard Reference Material should be stored in a well-closed container at room temperature (30 °C or less). It should not be subjected to heat or direct sunlight during storage. Under proper storage, experience at NBS indicates this material to be stable for at least 5 years. If the material purity degrades beyond the limits certified, purchasers will be notified by NBS. It is recommended that material not be used after 5 years from date of purchase.

There are several opinions in the literature regarding the stability of uric acid solutions [1,4,5,6,7,8]. It is recommended that the 1 mg/ml "stock" standard solution be considered stable for 3 months when stored in a well-stoppered, all-glass, dark-brown bottle at 4 °C. The 5 μg/ml "working" standard solution should be prepared fresh weekly and stored in the same manner.

References:

[1] Caraway, W. T., Uric Acid, in Standard Methods of Clinical Chemistry, Vol. 4, David Seligson, Editor-in-Chief, pp. 239-247, Academic Press, Inc., New York, N. Y. 1963.

[2] Natelson, S., Uric Acid, in Standard Methods of Clinical Chemistry, Vol. 1, Miriam Reiner, Editor-in-Chief, pp. ~~55-59~~ 123-135, Academic Press, Inc., New York, N. Y., 1953.

[3] Faulkner, W. R., and King, J. W., Renal Function Tests, in Fundamentals of Clinical Chemistry, N. W. Tietz, Editor, pp. 726-729, W. B. Saunders Company, Philadelphia, Pa. 1970.

[4] Feichtmeir, T., and Wrenn, H., Direct determination of uric acid using uricase. Am. J. Clin. Path. 25, 833-845 (1955).

[5] Dubbs, C., Davis, F. W., and Adams, W. S., Simple microdetermination of uric acid. J. Biol. Chem. 218, 497-504 (1956).

[6] Liddle, L., Seegmiller, J. E., and Laster, L., The enzymetric spectrophotometric method for determination of uric acid. J. Lab. and Clin. Med. 54, 903-913 (1959).

[7] Henry, R. J., Sobel, C., and Kim, J., A modified carbonate-phosphotungstate method for the determination of uric acid and comparison with the spectrophotometric uricase method. Am. J. Clin. Path. 28, 152-160 (1957).

[8] Henry, R. J., Clinical Chemistry, Principles and Technics, pp. 276-287. Hoeber Medical Division, Harper and Row, New York, N. Y. 1967.

This Standard Reference Material has been measured and certified at the Laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D. C. 20234

The date of issuance and certification of this Standard Reference Material was September 24, 1968.

U. S. Department of Commerce
Frederick B. Dent
Secretary

National Bureau of Standards
Richard W. Roberts, Director

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 914

CREATININE

This Standard Reference Material is certified as a chemical of known purity. It is intended primarily for use in the calibration and standardization of procedures for creatinine determinations employed in clinical analysis and for routine evaluations of daily working standards used in these procedures.

Purity	99.8 percent
Volatile matter	0.03 percent
Chloride	0.07 percent
Ash	0.003 percent
Insoluble matter	0.001 percent

The value of the purity has an estimated inaccuracy of 0.1 percent.

The creatinine used for this Standard Reference Material was obtained from the Pfanstiehl Laboratories, Inc., of Waukegan, Illinois. Analyses were performed by D. A. Becker, R. F. Brady, Jr., M. M. Darr, T. E. Gills, R. A. Paulson, W. P. Schmidt, and R. S. Tipson of the Analytical Chemistry Division.

The overall direction and coordination of technical measurements leading to the certification were under the chairmanship of R. Schaffer.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D. C. 20234
September 24, 1968
Revised November 23, 1973

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

The homogeneity of the creatinine was determined by paper, thin–layer, and gas–liquid chromatography.

Volatile matter was determined by measurement of loss of weight of the creatinine after heating for 24 hours at 110 °C.

Phase–solubility analysis [1] of an oven–dried sample of the Standard Reference Material, with absolute methanol as the solvent, indicated the purity to be 99.82 weight percent. Phase–solubility analysis of the undried creatinine with 95 percent methanol, and 95 percent ethanol, indicated purities of 99.81 and 99.76 weight percent, respectively. Potentiometric titration of the Standard Reference Material required 99.82 percent of the theoretical amount of hydrochloric acid.

The certified value for chloride was obtained by titration with mercuric nitrate of a solution of the products resulting from an oxygen–flask combustion of the Standard Reference Material

The ultraviolet absorption spectrum of a solution of the creatinine in water showed an extinction coefficient (ϵ_{max}) of 7140 ± 30 at 234 nm. The measure of uncertainty is the standard deviation of a single measurement, and should not be considered to be a certified measure of inaccuracy for the extinction coefficient. Infrared absorption and nuclear magnetic resonance spectra provided no evidence of impurities.

An emission spectrometric analysis for metallic constituents in the ash from this Standard Reference Material showed the following present as major constituents: silicon, aluminum, sodium, titanium, iron, and magnesium. Neutron activation analysis of the bulk Standard Reference Material indicated the presence of the following approximate concentrations of elements: chlorine, 330 ppm; copper, 0.2 ppm; manganese, 0.07 ppm; sodium, 30 ppm.

This Standard Reference Material is for "in vitro" diagnostic use only.

This material is intended for use as a standard in clinical chemistry. A "stock" standard solution containing 1 mg/ml of creatinine may be prepared by weighing 0.100 g of SRM 914 into an 100-ml volumetric flask. The flask is then filled nearly to the mark with 0.1N hydrochloric acid and agitated until solution is complete. The flask is then filled to the mark with 0.1N hydrochloric acid. This solution should be stored in a refrigerator. A "working" standard containing 20 μg/ml of creatinine may be prepared by diluting 2.0 ml of the "stock" standard solution to 100 ml with distilled water in a volumetric flask [2]. This "working" standard solution should be prepared daily.

This Standard Reference Material should be stored in a well-closed container at room temperature (30 °C or less). It should not be subjected to heat or direct sunlight during storage. Refrigerated storage is recommended. Under proper storage, experience at NBS indicates this material to be stable for at least 5 years. If the material degrades beyond the limits certified, purchasers will be notified by NBS. It is recommended that material not be used after 5 years from the date of purchase.

The "stock" standard solution containing 1 mg/ml, prepared as described above, is stable indefinitely when stored in a refrigerator at 4 °C in a well-stoppered, all-glass container. The dilute "working" standard solution should be prepared daily from the "stock" standard solution [3].

References:

[1] W. J. Madors, Phase–Solubility Analysis, in Organic Analysis, Vol. II, p. 253, Interscience Publishers, Inc., New York 1954.

[2] N. W. Tietz, Fundamentals of Clinical Chemistry, pp. 722-726, W. B. Saunders Co., Philadelphia, Pa. 1970.

[3] R. D. Henry, Clinical Chemistry, Principles and Technics, pp. 292-302, Hoeber Medical Division, Harper & Row, New York, N. Y. 1967.

This Standard Reference Material has been measured and certified at the laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D. C. 20234

The date of issuance and certification of this Standard Reference Material was September 24, 1968.

U. S. Department of Commerce
Frederick B. Dent
Secretary

National Bureau of Standards
Richard W. Roberts, Director

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 915

Calcium Carbonate

This Standard Reference Material is certified as a chemical of known purity. It is intended primarily for use in the calibration and standardization of procedures for calcium determinations employed in clinical analysis and for routine critical evaluation of the daily working standards used in these procedures.

The sample consists of highly purified calcium carbonate, and chemical assay as well as analysis for specific impurities indicate that the material may be considered to be essentially pure.

Purity	99.9+ percent
Water	0.01 ± 0.005 percent

Replicate samples taken from a randomly selected region of the undried material were assayed by a coulometric acidimetric procedure. The results from nine independent determinations, based on expression of the assay as calcium carbonate, indicate a purity of 99.99+percent with a standard deviation of 0.003 percent. Samples equilibrated at a relative humidity of 90 percent and assayed by this coulometric procedure showed a maximum moisture adsorption of 0.02 percent as compared to samples that were dried for 6 hours at 210 °C. The moisture content, similarly measured, on samples equilibrated at 75 percent relative humidity, was found to be 0.01 percent. The water content was determined by the Karl Fischer method.

The calcium carbonate used for this Standard Reference Material was obtained from the J. T. Baker Chemical Company, of Phillipsburg, New Jersey. Analyses were performed by C. E. Champion, E. R. Deardorff, G. Marinenko, O. Menis, T. C. Rains, T. A. Rush, W. P. Schmidt, B. F. Scribner, V. C. Stewart, J. K. Taylor, and D. W. Vomhof.

The overall direction and coordination of technical measurements leading to the certification were under the chairmanship of R. Schaffer.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by J. L. Hague.

Washington, D. C. 20234
March 4, 1969
Revised November 21, 1973

J. Paul Cali, Chief
Office of Standard Reference Materials

The material was examined for compliance with the specifications for reagent grade calcium carbonate as given in *Reagent Chemicals*, 4th edition, published by The American Chemical Society. The material was found to meet or exceed the minimum requirements in every respect. Examination by thermal gravimetric analysis indicated the loss of a minute proportion of weight below 175 °C (volatile matter) and the composition was stable above this temperature until a temperature of 625 °C, above which decomposition (evolution of CO_2) set in.

A semi-quantitative survey for trace contaminants by emission spectroscopy indicated the presence of less than 0.001 percent of copper, iron, magnesium, manganese and silicon in the material. By atomic absorption magnesium was evaluated at 1.0, sodium at 0.4, and strontium 2.1 parts per million (ppm); potassium was less than 0.4, lithium less than 0.05, and barium much less than 10 ppm. Neutron activation analysis indicated copper 0.9, manganese 0.6 and sodium 0.5 ppm. Copper was determined at 1 ppm by spectrophotometry.

This Standard Reference Material is intended for "in vitro" diagnostic use only.

This material is for use as a standard in clinical chemistry. It may be used to prepare calcium standard solutions for either atomic absorption or titrimetric methods of analysis.

Standard stock solutions of calcium for atomic absorption (Reference Method) [1]: Prepare a minimum of three concentrations at 2.00, 2.50, and 3.00 mmol of calcium per liter, with each to contain 140 mmol of sodium chloride and 5.0 mmol of potassium chloride per liter. To each of three 1-liter volumetric flasks, add 8.18 g NaCl and 373 mg of KCl. To the first flask (2.00 mmol of Ca per liter) add 200.2 mg of SRM 915 (dried at 200 °C for 4 hours), to the second flask (2.50 mmol of Ca per liter) add 250.2 mg of SRM 915, and to the third flask (3.00 mmol of Ca per liter) add 300.3 mg of SRM 915. To each flask add a few milliliters of water and 1 ml of concentrated HCl. Make sure that all the calcium carbonate is in solution before diluting with water up to the neck. When at ambient temperature, dilute each flask to the calibrated volume and mix by inverting the flask 30 times. Pipettings and dilutions of this material should follow the instructions given in reference [1].

Stock solution of calcium for titrimetric procedure: Place 0.250 g of dried SRM 915 into a 1000-ml volumetric flask. Add approximately 9 ml of deionized water and 1 ml concentrated HCl. Shake until dissolved. Fill to the mark with deionized water and store in a pyrex bottle. This solution contains 10.0 mg per 100 ml or 5.00 meq. per liter.

This Standard Reference Material should be stored in a well-stoppered bottle (preferably the original bottle) at room temperature. Calcium carbonate is a stable material. Under proper storage, experience at NBS indicates this material to be stable for at least 10 years. If the material purity degrades beyond the limits certified, purchasers will be notified by NBS. It is recommended that the material not be used after 5 years from the date of purchase.

Solutions prepared from SRM 915, Calcium Carbonate, are stable indefinitely when stored in an glass-stoppered bottle. All such solutions should be clear and display no turbidity.

References:

[1] J. P. Cali, G. N. Bowers, Jr., and D. S. Young, Clin. Chem. **19**, 1208-1213 (1973).

[2] N. W. Tietz, Fundamentals of Clinical Chemistry, pp. 636-648, W. B. Saunders Co., Philadelphia, Pa., 19105 (1970).

This Standard Reference Material has been measured and certified at the laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
B311, Chemistry Building
National Bureau of Standards
Washington, D. C. 20234

The date of issuance and certification of this Standard Reference Material was March 4, 1969.

U. S. Department of Commerce
Frederick B. Dent
Secretary

National Bureau of Standards
Richard W. Roberts, Director

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 916
Bilirubin

This standard reference material is certified as a chemical of known purity. It is intended primarily for use in the calibration and standardization of procedures for bilirubin determination employed in clinical analysis and for routine critical evaluation of the daily working standards used in these procedures.

Purity (bilirubin content)	99.0 percent
Chloroform	.0.8 percent
Insoluble (in chloroform)	less than 0.01 percent
Ash	0.01 percent

The value of the bilirubin content has a possible estimated inaccuracy of two percent. (See section on thin-layer chromatography).

The certification of this Standard Reference Material is based on the best current state-of-the-art knowledge. In spite of decades of work on bilirubin, it is still felt that much of its basic chemistry is incomplete.

The material in chloroform (ACS reagent containing ethanol) at 25.0 °C shows at 453 nm an absorptivity (±1 SD) of 104.6 ± 0.2 liter cm^{-1} g^{-1}. In methyl sulfoxide at 25.0 °C, the absorptivity (±1 SD) of the material at 453 nm is 107.7 ± 0.6 liter cm^{-1} g^{-1}.

It is recommended that bilirubin and its solutions be handled only under low-intensity incandescent light. The bilirubin should be stored under conditions that totally exclude light. Once removed from the ampoule, the vial should be replaced for storage in an air-tight container and stored at low temperature. The vial must be allowed to return to room temperature before opening.

The bilirubin used for this standard reference material was obtained from Pfanstiehl Laboratories, Inc., of Waukegan, Illinois. Analyses were performed by R. F. Brady, Jr., R. W. Burke, A. Cohen, B. Coxon, W. D. Dorko, D. Enagonio, A. J. Fatiadi, D. H. Freeman, J. M. Ives, B. A. Johnson, E. C. Kuehner, R. A. Paulson, T. C. Rains, R. Schaffer, W. P. Schmidt, J. T. Sterling, J. K. Taylor, B. F. West, W. J. Zielinski, Jr.

The overall direction and coordination of technical measurements leading to the certification were under the chairmanship of R. Schaffer.

The technical and support aspects concerning the preparation, certification, and issuance of this standard reference material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D. C. 20234
March 10, 1971

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

The standard reference material was prepared from material that was isolated from hog bile and crystallized as the acid. It was purified further by treatment in chloroform solution with sodium sulfate according to Fog [1] and recrystallization from chloroform.

Ash was determined on 100-mg samples heated overnight at 250 °C and then for 3 hrs at 650 °C. Flame photometry and atomic absorption measurements on a solution of the ash obtained from 0.275 g of the material showed, in μg/g (ppm) of metal: <0.05 lithium, 7.3 sodium, 1.6 potassium, 2.1 calcium, and 2.3 magnesium.

Elemental composition found for the material was: C, 67.36; H, 6.24; N, 9.42; Cl, 0.73. Required for a bilirubin preparation containing 0.8 percent of chloroform: C, 67.25; H, 6.17; N, 9.50; Cl, 0.71. In these analyses, nitrogen was determined by the Kjeldahl technique; and chlorine was determined by Carius digestion followed by gravimetric determination as silver chloride. By the latter method and recalculated as chloroform, the average (±1 SD) of ten 25-mg samples, was 0.83 ± 0.04 percent. The chloroform content was also obtained by heating at 250 °C at 0.5 torr for 4 hrs; the average (± 1 SD) of six 100-mg samples was 0.79 ± 0.02 percent. (Only about one-half or one-tenth of the chloroform was removed at 180 °C and at 110 °C, respectively.) During gradual heating of the material in a quadrupole mass spectrometer, only ion-fragments characteristic of chloroform were observed until the sample was heated above 300 °C, whereupon water and carbon dioxide were detected, and at a somewhat higher temperature, the mass spectrum typical of bilirubin was obtained. Gas chromatography and nuclear magnetic resonance spectroscopy with the material dissolved in methyl sulfoxide confirmed the presence of chloroform. Because the chloroform is so firmly retained and homogeneously dispersed, it is recommended that the material be used as supplied.

Thermogravimetric analysis with the sample heated in a dry nitrogen atmosphere at 5 °C/min showed the initiation of the loss of a large proportion of sample weight at temperatures between 319 and 323 °C; determined in air, however, comparable weight-loss began between 288 and 292 °C.

For determining the absorptivity of the standard material, absorbances were recorded with a Cary 16 spectrophotometer on solutions using a capped 1.000 ± 0.001-cm path-length cuvette in a cell-holder thermostated at 25.0 ± 0.1 °C. Solution preparation and handling were performed under low intensity, incandescent light; at other times, solutions were totally protected from light. To preclude any change in concentration of chloroform solutions due to evaporation of the solvent during solution transfer, nitrogen gas was used to force the solution from the flask through Teflon tubing into the cuvette and, to effect rinsing of the cuvette, from the latter to an overflow. Solutions in methyl sulfoxide were transferred to the cuvette by pipet.

For the determination of chloroform-insoluble matter, sample (0.1 g) and solvent (200 ml), protected from light, were agitated in an ultrasonic bath, heated to 60 °C briefly, and then cooled and filtered, using pressure, (with 50 ml of the solvent for washing) through a tared 0.4 μm-porosity membrane.

<u>Extraction by Bicarbonate.</u> Yellow extracts were obtained by shaking a solution of the standard reference material (0.010 g) in chloroform (40 ml) with 10-ml volumes of 0.6 molar aqueous sodium bicarbonate. Their absorption spectra, with maxima in the range 415--430 nm, showed absorbance values that ranged from 0.035 to 0.030 in successive extracts using 1-cm cells. The qualitative and quantitative compositions of such extracts were examined using the filtered chloroform solutions containing 0.1-g quantities of the standard reference material that were prepared in the tests for chloroform insolubles: Extractions twice with 50-ml volumes of the bicarbonate solution and with water, and neutralization of the combined aqueous extracts followed by extraction into chloroform, yielded pigment (dry weight approximately 0.2 mg) that was shown by its spectrum and specific absorptivity to be typical of bilirubin in chloroform. Thus, bicarbonate extraction provided no evidence of impurities more acidic than bilirubin.

Extraction by Carbonate. Extraction of a 200-ml chloroform solution containing 0.1 g of standard with 100-ml volumes of 0.1 molar sodium carbonate reduced the 1-cm absorbance value of the chloroform layer to approximately 0.22 after the second extraction, and to 0.08 and then 0.01 with the third and fourth extractions, respectively. On washing with water and then evaporating the extracted chloroform layer to dryness, residue weights were typically about 0.01 mg. Hence, this extraction process showed a negligible proportion of nonacidic impurities.

Thin-layer Chromatography. An impurity in the standard material may be detected by thin-layer chromatography with polyamide as the absorbent and 3:1 (v/v) methanol--aqueous ammonium hydroxide (3.3 percent) as the developer [2]. Thus, 0.05 mg of the material (spotted from a chloroform solution) provides, on development, an elongated, yellow-orange streak preceded (and usually separate) by a faintly-visible, yellow spot. Under 366-nm irradiation, a pink fluorescence develops very quickly at the location of this spot. The material responsible for the pink fluorescence and its precursor have not been characterized. A means for obtaining a reliable estimate of this contribution to overall impurity has not been ascertained, but is under study. Repurification of the standard material does not affect this behavior.

This Standard Reference Material is intended for "in vitro" diagnostic use only.

This material is for use as a standard in clinical chemistry. A standard solution for the development of a calibration curve may be obtained as follows. Weigh out exactly 20.2 mg of SRM 916, transfer to a 100-ml volumetric flask, and dissolve in 2 ml of 0.1M sodium carbonate and 1.5 ml of 0.1N sodium hydroxide. The solution should be clear and red in color. Dilute this solution to 100 ml with "acceptable" [3], pooled serum and mix well. Since bilirubin is light sensitive, the flask should be wrapped in aluminum foil and kept as dark as possible. It is further recommended that the solution preparation and handling be carried out under low-intensity incandescent light [3,4].

Standard solutions of lower concentration may be prepared by dilution of appropriate aliquots of the above solution with "acceptable" pooled serum.

Standard Reference Material 916 is supplied in a vial, which is sealed in an ampoule under argon. The sample must be protected from light. After opening, the material should be kept in the tightly-closed vial in a desiccator, protected from light by a black plastic bag. Refrigeration at 4 °C is suggested. Under proper storage, experience at NBS indicates this material to be stable for at least 3 years. If the material degrades beyond the limits certified, purchasers will be notified by NBS. It is recommended that this material not be used after 3 years from the date of purchase.

Solutions of bilirubin, prepared as described above, may be preserved for about a week under refrigeration at –20 °C.

References

[1] Fog, J., Scand. J. Clin. Lab. Invest., 16, 49 (1964).

[2] Petryka, Z. J., and Watson, C. J., J. Chromatog., 37, 76 (1964).

[3] Recommendation on a uniform bilirubin standard, Clin. Chem. 8, 405-407 (1962).

[4] Tietz, N. W., Fundamentals of Clinical Chemistry, W. B. Saunders Co., Philadelphia, Pa., 1970, pp. 755-762.

This Standard Reference Material has been measured and certified at the laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D. C. 20234

The date of issuance and certification of this Standard Reference Material was March 10, 1971.

U. S. Department of Commerce
Frederick B. Dent
Secretary
National Bureau of Standards
Richard W. Roberts, Director

Certificate of Analysis
National Bureau of Standards

Standard Reference Material 917
D-Glucose (Dextrose)

B. Coxon and R. Schaffer

This standard reference material is certified as a chemical of known purity. It is intended primarily for use in the calibration and standardization of procedures for glucose determinations employed in clinical analysis and for routine critical evaluation of the daily working standards used in these procedures.

Purity	99.9	percent
α-D-Glucopyranose	greater than 99.0	percent
β-D-Glucopyranose	less than 1.0	percent
Moisture	0.06	percent
Ash	0.002	percent
Insoluble Matter	0.001 to 0.006	percent
Nitrogen	less than 0.001	percent

Specific Rotation

$[\alpha]_{D}^{20}$ = +53.2 ° (at equil., *c* 20.1 in water)

$[\alpha]_{546}^{20}$ = +62.8 ° (at equil., *c* 20.1 in water)

$[\alpha]_{D}^{20}$ = +112.6 ° (initial, *c* 10.05 in methyl sulfoxide)

The value for the purity has an estimated inaccuracy of ±0.1 percent.

The D-glucose used for this standard reference material was obtained from Pfanstiehl Laboratories, Inc., of Waukegan, Illinois. Analyses were performed by R. F. Brady, Jr., B. Coxon, M. M. Darr, T. E. Gills, E. C. Kuehner, R. A. Paulson, T. C. Rains, T. A. Rush, W. P. Schmidt, J. H. Thomas, and W. L. Zielinski of the Analytical Chemistry Division.

The overall direction and coordination of technical measurements leading to the certification were under the chairmanship of R. Schaffer.

The technical and support aspects concerning the preparation, certification, and issuance of this standard reference material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D. C. 20234
November 18, 1970
Revised September 20, 1973

J. Paul Cali, Chief
Office of Standard Reference Materials

The only impurities detected in this standard reference material were moisture and traces of inorganic compounds. Paper, thin-layer, and high-pressure ion-exchange chromatographic techniques revealed no organic impurities.

The proportion of the α-D-glucose anomer was estimated by three methods. The ratio of the β-anomer to the α-anomer was found to be 0.5:100 by gas-liquid chromatography (glc) after per(trimethylsilyl)ation of the solid standard material for 10 min at 0 °C using N-(trimethylsilyl)imidazole in anhydrous pyridine to minimize possible mutarotation. Use of this glc technique on partly melted standard material showed that the proportion of the β-anomer increased markedly during melting. Differential scanning calorimetry of the standard material, contained under nitrogen in unsealed aluminum pans, showed the α-D-glucose content to be 99.4 percent. This value represents only the proportion of anhydrous α-D-glucopyranose present, since the method treats β-D-glucose, or hydrates of α-D-glucose that are stable up to the melting point, as impurities. However, proton magnetic resonance spectroscopy at 90 MHz indicated the ratio of β-anomer to α-anomer to be 0.9:100. This determination was performed 10 min after dissolution of 100 mg of D-glucose in 0.5 ml of methyl sulfoxide-d_6, by integration of the doublets due to the anomeric hydroxyl groups.

Optical rotations were obtained by use of an automatic polarimeter and a high-precision manual polarimeter.

The moisture content reported was determined by the Karl Fischer and near infrared methods. As only 0.01 to 0.02 percent in weight was lost on drying at 70 °C/1-2 torr for 100 hr, the analyses reported herein were performed on the undried standard reference material.

The ash content reported was determined by ignition of 20-g samples at 750 °C; the undissolved residue, on 10-g samples. Turbidimetric assays of solutions of the standard showed the presence of chloride at 2 ppm and sulfate at 3 ppm. Neutron activation indicated chloride at 4 ppm.

Emission spectrometric analysis of the ash from this standard reference material showed calcium to be approximately 5 ppm; magnesium and silicon each less than 0.1 ppm; aluminum, boron and iron each less than 0.05 ppm; and copper, less than 0.01 ppm.

Atomic absorption spectrometry of the standard reference material indicated that it contains less than 0.5 ppm of magnesium. Flame emission spectrometry indicated the content of calcium to be 1 ±0.5 ppm, and sodium to be 2.9 ppm.

This Standard Reference Material is intended for "in vitro" diagnostic use only.

This material is for use as a standard in clinical chemistry. A 1 percent standard solution of glucose may be prepared by weighing 1.000g of SRM 917 into an 100-ml volumetric flask. The flask is then filled nearly to the mark with 0.2 percent benzoic acid solution and agitated until solution is complete. The flask is then filled to the mark with 0.2 percent benzoic acid solution. One ml of this solution contains 0.01g (10mg) of glucose. The benzoic acid solution should be prepared from ACS Reagent grade benzoic acid. Appropriate size samples of this solution are analyzed by the exact procedure used for the submitted specimen of body fluid.

To prepare glucose solution of lower concentration the appropriate aliquot is pipetted into a 100-ml volumetric flask and diluted with 0.2 percent benzoic acid solution.

This Standard Reference Material should be stored in a well-closed container at room temperature (30 °C or less). It should not be subjected to heat or direct sunlight during storage. Refrigerated storage is recommended. Under proper storage, experience at NBS indicates this material to be stable for at least 5 years. If the material purity degrades beyond the limits certified, purchasers will be notified by NBS. It is recommended that material not be used after 5 years from date of purchase.

The 1 percent standard glucose solution, prepared as described above, is stable indefinitely when stored in a refrigerator at 4 °C and in a well-stoppered, all-glass container. The dilute glucose standard should be prepared daily from the 1 percent stock solution [1,2].

All constituted solutions of D-Glucose should be clear and without indications of bacterial growth of any kind.

References:

[1] R. D. Henry, Clinical Chemistry, Principles and Practice, pp. 625-656, Hoeber Medical Division, Harper & Row, New York, New York 10016 (1967).

[2] N. W. Tietz, Fundamentals of Clinical Chemistry, pp. 154-163, W. B. Saunders Co., Philadelphia, Pa. 19105 (1970).

This Standard Reference Material has been measured and certified at the Laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D. C. 20234

The date of issuance and certification of this Standard Reference Material was November 18, 1970.

U. S. Department of Commerce
Frederick B. Dent
Secretary
National Bureau of Standards
Richard W. Roberts, Director

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 918

Potassium Chloride

(Clinical Standard)

This standard reference material is certified as a chemical of known purity. It is intended primarily for use in the calibration and standardization of procedures employed in the determination of potassium and chloride ions in clinical analyses. The sample consists of highly purified potassium chloride. Chemical assay as well as analyses for specific impurities indicate that the material may be considered essentially pure, except for moisture due to occlusion.

Purity . . . . 99.9 ± 0.0 percent

The above value for the purity of the material is based on a sample dried over magnesium perchlorate for 24 hours. Potassium chloride is hygroscopic when the relative humidity at room temperature exceeds 75 percent, but can be dried to the original weight by desiccation over freshly exposed P_2O_5 or $Mg(ClO_4)_2$ for 24 hours. The material should be stored with such a desiccant. The potassium was determined by a combination of gravimetric and isotope dilution analyses. More than 99 percent of the potassium was precipitated, filtered, and weighed as potassium perchlorate. The weight of potassium perchlorate was corrected for rubidium perchlorate. The soluble potassium was determined by isotope dilution mass spectrometry. Total potassium was the sum of the potassium from the potassium perchlorate and the potassium from the filtrate. The chloride was determined by a coulometric argentimetric procedure.

Based on 12 independent measurements for each ion, the sample was considered homogeneous. Material dried at 500 °C for 4 hours in a platinum or Vycor crucible (Pyrex is unsatisfactory) was assayed at 99.98 ± 0.01 percent. The loss of moisture by this procedure was about 0.07 percent.

The potassium chloride used for this standard reference material was obtained from the J. T. Baker Chemical Company, of Phillipsburg, New Jersey. Analyses were performed by G. Marinenko, T. J. Murphy, T. C. Rains, T. A. Rush, W. P. Schmidt, and V. C. Stewart.

The overall direction and coordination of technical measurements leading to the certification were under the chairmanship of W. R. Shields.

The technical and support aspects concerning the preparation, certification, and issuance of this standard reference material were coordinated through the Office of Standard Reference Materials by J. L. Hague.

Washington, D. C. 20234
January 22, 1971
Revised November 23, 1973

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

This material was examined for compliance with the specifications for reagent grade potassium chloride as given in Reagent Chemicals, 4th edition, published by the American Chemical Society. The material met or exceeded the minimum requirements in every respect.

A semi-quantitative survey for trace contaminants by emission spectroscopy indicated the presence of less than 0.001 percent aluminum, copper, iron, and magnesium. A value of 0.24 parts per million (ppm) of magnesium was obtained by atomic absorption spectrometry. Flame emission spectrometry indicated the presence of the following elements: rubidium, 27 ppm; sodium, 9 ppm; lithium, 0.6 ppm; and cesium and calcium, less than 2 ppm.

This Standard Reference Material is intended for "in vitro" diagnostic use only.

This material is intended for use as a standard for potassium and, to a lesser degree, for chloride determination in clinical chemistry.

Potassium is most frequently determined by flame emission photometry. The operative details of this methodology vary from instrument to instrument and are discussed at length in their respective operating manuals. A standard solution of potassium chloride (10 mmol potassium per liter) may be prepared as follows: transfer 0.746 g of SRM 918 that has been dried to constant weight quantitatively to a 1-liter volumetric flask, dilute to volume with water, and make the solution uniform by inverting the flask at least 30 times. The concentrations required for analysis may be prepared by accurate dilution of this standard with deionized water.

A standard solution of chloride containing 100 mmol per liter may be prepared by transferring quantitatively 7.46 g of SRM 918 to a 1-liter volumetric flask and adding 3 ml of concentrated nitric acid (ACS Reagent Grade) and 100 ml of deionized water. After all the salt is dissolved, dilute to volume with deionized water. It should be noted that a chloride standard solution prepared from sodium chloride contains the respective ions in a ratio more nearly that of normal serum than a solution prepared from potassium chloride.

This Standard Reference Material should be stored in the well-closed original container under normal laboratory conditions. It is recommended that weighing and other manipulations of the solid SRM not be made when the relative humidity exceeds 75 percent.

The solutions of SRM 918 are stable indefinitely when stored in a well-stoppered, all-glass container. All such solutions should be clear and display no turbidity.

References:

[1] P. M. Hald and W. B. Mason, "Sodium and potassium by flame photometry", in Standard Methods of Clinical Chemistry, Vol. 2, David Seligson, editor-in-chief, pp. 165-185, Academic Press, Inc., New York, (1958).

[2] N. W. Tietz, Fundamentals of Clinical Chemistry, pp. 621-625, W. B. Saunders Company, Philadelphia, Pa. (1970).

This Standard Reference Material has been measured and certified at the laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
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The date of issuance and certification of this Standard Reference Material was January 22, 1971.

U. S. Department of Commerce
Frederick B. Dent
Secretary

National Bureau of Standards
Richard W. Roberts, Director

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 919
Sodium Chloride
(Clinical Standard)

This Standard Reference Material is certified as a chemical of known purity. It is intended primarily for use in the calibration and standardization of procedures employed in the determination of sodium and chloride ions in clinical analyses. The sample consists of highly purified sodium chloride. Chemical assay as well as analyses for specific impurities indicate that the material may be considered essentially pure, except for occluded moisture.

Purity . . . . 99.9 ± 0.0 percent

The above value for the purity of the material is based on a sample dried over magnesium perchlorate for 24 hours. At room temperature sodium chloride is hygroscopic above 60 percent relative humidity. The sorbed water can be removed, however, by desiccation over freshly exposed P_2O_5 or $Mg(ClO_4)_2$ for 24 hours. Chloride was determined using the coulometric method of Marinenko and Taylor [J. Res. NBS, 67A, 31(1963)].

Based on 8 independent measurements of chloride content, the sample is considered homogeneous.

When the material is crushed and dried at 200 °C for 18 hours, the loss of moisture is about 0.08 percent. Coulometric determinations of chloride on the dried material indicate 99.995 ± 0.004 percent purity.

The sodium chloride used for this Standard Reference Material was obtained from the J. T. Baker Chemical Company, of Phillipsburg, New Jersey. Analyses were performed by G. Marinenko, J. R. Baldwin, M. Darr, and T. C. Rains.

The overall direction and coordination of technical measurements leading to the certification were under the chairmanship of R. A. Durst.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D. C. 20234
August 6, 1973
Revised November 23, 1973

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

This material was examined for compliance with the specifications for reagent grade sodium chloride as given in Reagent Chemicals, 4th edition, published by the American Chemical Society. The material meets or exceeds the minimum requirements in every respect.

Sodium was assayed using a gravimetric procedure in which the sodium chloride was converted to sodium sulfate. Approximately 250 mg of sodium chloride (dried at 500 °C for 4 hours in a platinum crucible) was dissolved in ultrapure sulfuric acid solution (1:1) and evaporated to dryness. Ammonium carbonate was added and the crucible slowly heated to 600 °C, then 900 °. This treatment was repeated until the weight of sodium sulfate remained constant. Based on 6 determinations, the sodium assay is 39.3_2 weight percent or 99.9_6 percent of the amount computed for perfectly pure, stoichiometric NaCl.

A semiquantitative survey for trace elements by emission spectroscopy indicated less than 10 μg/g calcium, copper, iron, and magnesium. A value of less than 3 μg/g magnesium was obtained by atomic absorption spectrometry. Flame emission spectrometry indicated the presence of the following elements: potassium, 11 μg/g; calcium and cesium, less than 2 μg/g; and rubidium and lithium less than 0.5 μg/g.

This Standard Reference Material is intended for "in vitro" diagnostic use only.

This material is intended for use as a standard for the determination of sodium and chloride ions in clinical chemistry.

Sodium is most frequently determined by flame emission photometry. The operative details of this methodology vary from instrument to instrument and are discussed at length in their respective operating manuals. A standard solution of 100 mmol of sodium chloride per liter (suitable for both sodium and chloride determinations) may be prepared by placing 5.85 g of SRM 919 (dried at 110 °C) in a 1-liter volumetric flask and adding 3 ml of concentrated nitric acid (ACS Reagent Grade) and 100 ml of deionized water. After the NaCl is dissolved, dilute to the mark with deionized water. The concentration required for analysis may be prepared by accurate dilutions with distilled water.

This Standard Reference Material should be stored in the well-closed original, bottle under normal laboratory conditions. It is recommended that weighing and other manipulations not be made when the relative humidity exceeds 60 percent.

Solutions of SRM 919 are stable indefinitely when stored in a well-stoppered, all-glass container. All such solutions should be clear and display no turbidity.

References:

[1] N. W. Tietz, Fundamentals of Clinical Chemistry, pp. 616-618; 621-625, W. B. Saunders Co., Philadelphia, Pa. (1970).

[2] R. D. Henry, Clinical Chemistry, Principles and Practice, pp. 345-350: 402-409, Hoeber Medical Division, Harper & Row, New York, N. Y. (1967).

This Standard Reference Material has been measured and certified at the laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D. C. 20234

The date of issuance and certification of this Standard Reference Material was August 6, 1973.

U. S. Department of Commerce
Frederick B. Dent
Secretary

National Bureau of Standards
Richard W. Roberts, Director

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 920
D-Mannitol

B. Coxon and R. Schaffer

This Standard Reference Material is certified as a chemical of known purity. It is intended primarily for use in the calibration and standardization of procedures for triglycerides determinations employed in clinical analysis and for routine critical evaluation of the daily working standards used in these procedures.

D-Mannitol	99.8 percent
D-Glucitol	0.1 percent
Total Alditol	99.9 percent
Loss on drying	<0.02 percent
Ash	<0.001 percent
Insoluble matter	<0.001 percent

The value for the purity has an estimated inaccuracy of 0.1 percent.

The D-mannitol used for this Standard Reference Material was obtained from Pfanstiehl Laboratories, Inc., of Waukegan, Illinois. Analyses were performed by R. F. Brady, Jr., B. Coxon, B. A. Johnson, W. H. McCurdy, and W. P. Schmidt of the NBS Analytical Chemistry Division.

The overall direction and coordination of technical measurements leading to the certification was under the chairmanship of R. Schaffer.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D. C. 20234
January 17, 1972
Revised November 23, 1973

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

The homogeneity of this Standard Reference Material was monitored by differential scanning calorimetry of 20 samples taken at random from the bulk material, and by polarimetry, gas-liquid chromatography, and thin-layer chromatography. Elemental microanalysis (±2 SD) showed carbon 39.51 ± 0.10 and hydrogen 7.86 ± 0.12; the calculated values are 39.56 and 7.75 percent, respectively. The Standard Reference Material melted at 167.0–168.0 °C (corr.) when heated in an open capillary at 0.5 $°C{\cdot}min^{-1}$.

The content of D-glucitol in the material was determined by gas-liquid chromatography of hexakis-*o*-(trifluoroacetyl) derivatives on a 10-ft. column of Gas Chrom Q (100-120 mesh) coated with 3 percent of GE-XE-60 liquid phase, using a flame-ionization detector. The derivatives were prepared by treating 200 mg of the D-mannitol with 2 ml of trifluoroacetic anhydride and 10 mg of sodium trifluoroacetate in 2 ml of nitromethane for 2 hours at 25-30 °C. The solution resulting was diluted to 5.0 ml with nitromethane, and 2-ml aliquots were injected into the gas chromatograph (inlet temperature, 135 °C; column, 145 °C; detector, 150 °).

The purity of the Standard Reference Material, as determined by differential scanning calorimetry, is 99.9 mole percent, and by phase solubility analysis, 99.8 weight percent. For differential scanning calorimetry, the D-mannitol (~0.5 mg) was heated from 380 to 436 K at 80 $K{\cdot}min^{-1}$ and then from 436 to 442 K at 0.625 $K{\cdot}min^{-1}$. Phase solubility analysis was conducted by equilibrating the material with either ethanol-water (1:1 v/v), or methanol-water (1:1 v/v) at 25 °C for six days.

No impurities were revealed by paper and thin-layer chromatography, infrared spectroscopy (KCl pellet), ultraviolet spectrophotometry (20-percent solution of the material in water), or by proton and carbon-13 magnetic resonance spectroscopy.

The proton magnetic resonance spectrum obtained at 90 MHz by accumulating 324 frequency sweeps of a solution of 0.1 g of the material in 0.5 ml of methyl sulfoxide-d_6 showed, as expected for D-mannitol, only a complex multiplet and a doublet at 4.4 and 4.2 ppm, respectively, from tetramethylsilane, due to six hydroxyl protons, and a complex multiplet at 3.5 ppm due to eight CH protons.

The proton-decoupled carbon-13 magnetic resonance spectrum, accumulated at 22.6 MHz from 1024 scans of a saturated solution of the material in water-hexafluoroacetone sesquihydrate (5:1 v/v), with heteronuclear, field-frequency stabilization on fluorine, showed only three singlets, at 122, 123, and 129 ppm from external, ^{13}C-enriched carbon disulfide, owing to the two-fold axis of symmetry of D-mannitol.

In the absence of proton decoupling, 5650 scans of a similar solution gave a carbon-13 nmr spectrum that showed only two overlapping doublets ($J13_{C\text{-}H}$ 146 and 141 Hz), each overlapped by a triplet ($J13_{C\text{-}H}$ 142 Hz).

Optical rotations were measured by means of an automatic polarimeter and a high-precision, manual polarimeter, by using solutions of the material either in water, or in aqueous 4.7 percent ammonium molybdate solution (20.0 ml) diluted to 25.0 ml with 0.5 M sulfuric acid at 20 °C.

$[\alpha]^{20}_{D} = 139.0°$ (*c* 1.6 in acidified 3.8 percent ammonium molybdate solution)

$[\alpha]^{20}_{D} = -0.3°$ (*c* 16.0 in water)

On being heated at 70 °C under vacuum for 24 h, 7-g samples of the Standard Reference Material lost 0.001 to 0.002 percent of their weight; however, at 110 °C and 30 torr, constant weight was not reached during 80 h, at which time, 50-g samples of the material had decreased in weight by 0.02 percent, corresponding mainly to sublimation of the samples.

The residue insoluble in water and the ash content of the Standard Reference Material were each determined on 25-g samples.

This Standard Reference Material is intended for "in vitro" diagnostic use.

This material is for use as a standard in the determination of triglycerides in clinical chemistry. Since mannitol solutions require no preliminary handling prior to the automated part of the analysis they are useful in checking automated systems. A stock solution may be prepared by transfer of 182.2 mg of SRM 920 to a 1-liter volumetric flask, dissolving it in 0.2 N sulfuric acid and diluting to the mark with 0.2 N sulfuric acid. This solution contains 1 μmol/ml of mannitol. Working solutions of 0.050, 0.100, and 0.150 μmol/ml may be prepared by dilution of appropriate aliquots with 0.2 N sulfuric acid [1,2].

This Standard Reference Material should be stored in a well-closed container at room temperature (30 °C or less). It should not be subjected to heat or direct sunlight during storage. Under proper storage, experience at NBS indicates this material to be stable for at least 5 years. If the material degrades beyond the limits certified, purchasers will be notified by NBS. It is recommended that material not be used after 5 years from date of purchase.

The stock standard solution (1 μmol/ml) and the working solutions should be stored in a glass-stoppered, brown bottle at 4 °C. Under such conditions these standards should be stable for 6 months.

All constituted solutions of D-mannitol should be clear and display no turbidity.

References:

[1] Kanter, S. L., Mannitol as a primary standard in the determinations of triglycerides. Clin. Chim. Acta 16, 177-178 (1967).

[2] Communication from Lipids Laboratory, Center for Disease Control, Atlanta, Ga., January 8, 1974.

This Standard Reference Material has been measured and certified at the laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D. C. 20234

The date of issuance and certification of this Standard Reference Material was Janaury 17, 1972.

U. S. Department of Commerce
Frederick B. Dent
Secretary

National Bureau of Standards
Richard W. Roberts, Director

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 921

Cortisol (Hydrocortisone)

B. Coxon and R. Schaffer

This Standard Reference Material is certified as a chemical of known purity. It is intended primarily for use in the calibration and standardization of procedures for cortisol determinations employed in clinical analysis and for routine critical evaluation of the daily working standards used in these procedures.

Constituent	Percent
Cortisol	98.9
21-Dehydrocortisol	0.6
21-O-Acetylcortisol	0.2
21-Dehydrocortisone	0.1
Cortisone	0.1
Total Steroids	99.9
Ash	0.002
Insoluble matter	0.001
Loss on drying	0.08

The cortisol assay has an estimated inaccuracy of 0.2 percent.

The cortisol used for this Standard Reference Material was obtained from the Upjohn Company, Kalamazoo, Michigan. Analyses were performed by R. F. Brady, Jr., A. Cohen, B. Coxon, M. Darr, W. D. Dorko, D. P. Enagonio, T. E. Gills, E. E. Hughes, W. P. Schmidt, and S. A. Wicks of the Analytical Chemistry Division.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D. C. 20234
February 15, 1973
Revised December 16, 1973

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

Identification and quantitation of the four steroid impurities were accomplished by Fourier-transform proton-magnetic-resonance (pmr) spectroscopy and thin-layer chromatography (tlc) performed on fractions from the liquid chromatography of 100 mg of the material in ethanol on a column (91 x 0.8 cm) of poly(vinylpyridine) crosslinked with 8 percent of divinylbenzene. Fractions were eluted with ethanol at a pressure of 4.7 kg·cm^{-2}. Tlc of aliquots of the fractions was performed on silica gel GF_{254} using 9:1 (v/v) chloroform-methanol. Equivalent sensitivity of detection was obtained by fluorescence quenching and by spraying with aqueous 20-percent sulfuric acid and heating at 120 °C. All the steroid impurities, except 21-dehydrocortisone, showed similar sensitivity. 4-Androsten-11β-ol-3,17-dione was found in liquid chromatography fractions, but could not be demonstrated directly by tlc of up to 1 mg of the bulk material. Because this compound was readily resolved from mixtures prepared with it and the bulk material and detected in proportion to amounts used for reference, the compound was adjudged an artifact produced during liquid chromatography. On the other hand, the major impurity, 21-dehydrocortisol, did not arise by the known copper-catalyzed oxidation of cortisol, as shown by its unaltered proportion by tlc of the bulk material even after EDTA-treatment of the system to remove copper.

The quantitative proportion of each steroid present in the sample was estimated from the dry weight (W) of the residue of each liquid chromatographic fraction and the measured intensity of the Fourier-transform generated methyl signals characteristic for each steroid, using the expression:

$$m_n M_n = \frac{W h_n M_n}{h_1 M_1 + h_2 M_2 \ldots + h_m M_m}$$

where m_n and M_n are the number of moles present and the molecular weight, respectively of the n_{th} component in a mixture of m components, h_n is the methyl signal intensity of the n_{th} component, and $h_1 \ldots h_m$ are the corresponding intensities of components 1...m, obtained by measurement of the methyl peaks above the methylene envelope of the steroids.

For proof of homogeneity, nine samples were withdrawn from the bulk Standard Reference Material according to a statistical plan. They were analyzed in a commercial carbon-hydrogen-nitrogen microanalyzer and were found to be homogeneous with respect to carbon and hydrogen content within the limits of precision of the method. Solutions of the samples in 95 percent ethanol at 25 °C showed an absorbance maximum at 242 nm with a molar extinction coefficient of 16.1 x 10^3 liter·cm^{-1}.mol^{-1}.

Elemental macroanalysis of the material showed carbon 69.49 ± 0.10 percent (2 SD of the mean); and hydrogen 8.39 ± 0.05 percent (2 SD of the mean); the calculated values for cortisol are 69.58 percent and 8.34 percent, respectively.

The Standard Reference Material melted at 219.0-220.5 °C (corrected) when heated in an open capillary tube at 0.5 °C·min^{-1}. The resulting pale yellow melt did not solidify on cooling. After sealing in a capillary tube under vacuum, the material melted at 220.5-221.5 °C without yellowing, but did not resolidify.

Thermogravimetric analysis of samples heated under dry nitrogen at 2 °C·min^{-1} showed the initiation of loss of a large proportion of sample weight at 221 °C (uncorrected). However, for samples heated in air, loss of weight began at 204 °C. The attempted application of differential scanning calorimetry to samples heated under nitrogen gave thermograms that were very dependent on the rate and time of heating, and that were not reproducible.

The mass spectrum of the Standard Reference Material obtained by electron induced ionization at 70eV and a probe temperature of 220 °C showed strong ion currents at *m/e* 362 [$M^{+\cdot}$], 344 [M–18(H_2O)], 332 [M–30(CH_2O)], 303 [M–59($COCH_2OH$)], 302 [M–60(CH_2=C=O and H_2O)], 285 [M–59–18], 42 [CH_2=C=O], and 31 [CH_2=O–H]$^+$. The peaks at *m/e* 302 and 163 correspond to the molecular ion and key fragment, respectively, of the thermal degradation product of cortisol, namely 4-Androsten-11β-ol-3,17-dione.

Optical rotations of solutions of the Standard Reference Material in ethanol (c 1.00) were measured at 20 °C by means of automatic and high-precision, manual polarimeters and are as follows:

	$[\alpha]_{\lambda}^{20}$ (±2 SD of the mean)	
λ (nm)	degrees	radians
589	168.9±0.1	2.948±0.002
578	177.3±0.1	3.094±0.002
546	205.6±0.1	3.588±0.002
436	387.9±0.4	6.770±0.007
365	571.7±0.6	9.978±0.010

Insoluble matter was determined by filtration of solutions of the Standard Reference Material (1.8-2.0 g) in methyl sulfoxide (9-20 ml) through Millipore filters (1.5 μm).

The loss of weight on drying was determined after heating three samples (2.8 g, each) of the material at 110 °C and 0.1 torr for 24 hours. No further weight loss occurred during heating of the samples for an additional 48 hours.

Ash was determined by heating of samples (10 g) of the Standard Reference Material to 700 °C. Spectrometric analysis of the ash indicated calcium as a major constituent, and sodium, magnesium, and silicon as minor constituents.

Destructive neutron activation analysis using SRM 1577, Bovine Liver, as a standard indicated that the Cortisol Standard Reference Material contains 0.035±0.005 ppm (±2 SD of the mean) of copper.

This Standard Reference Material is for "in vitro" diagnostic use only.

A standard solution containing 5 μg per ml of cortisol may be prepared as follows. Transfer 50 mg of SRM 921 to a 50-ml volumetric flask and add 35 ml of absolute ethanol. When the cortisol is completely dissolved, dilute to the mark with absolute ethanol. Dilute 1 ml of this ethanol solution to 200 ml with distilled water [1,2].

Working standard solution may be made by appropriate dilution of aliquots of the 5 μg per ml solution with distilled water.

This Standard Reference Material should be stored in a well-closed container at room temperature (30 °C or less). It should not be subjected to heat or direct sunlight during storage. Refrigerated storage is recommended. Under proper storage, experience at NBS indicates purified cortisol to be stable for at least 5 years. If the material purity degrades beyond the limits certified, purchasers will be notified by NBS. It is recommended that material not be used after 5 years from date of purchase.

The solution of 1 mg/ml of cortisol in ethanol as prepared above should be stored in a well-stoppered, all-glass container and kept in a refrigerator at 4 °C. The aqueous solution of 5μg per ml cortisol should also be stored in a well-stoppered, all-glass container at 4°C. Under these conditions both solutions should be stable for six months. The more dilute "working" standard solutions should be prepared daily.

All constituted solutions of cortisol should be clear and display no turbidity.

References:

[1] R. E. Peterson, "Hydrocortisone in plasma", in Standard Methods of Clinical Chemistry, Vol. 3, David Seligson, Editor-in-Chief, pp. 160-166, Academic Press, Inc., New York, N. Y. (1961).

[2] N. W. Tietz, Fundamentals of Clinical Chemistry, pp. 509-512. W. B. Saunders Company, Philadelphia, Pa., (1970).

This Standard Reference Material has been measured and certified at the laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D. C. 20234

The date of issuance and certification of this Standard Reference Material was February 15, 1973.

U.S. Department of Commerce
Elliot L. Richardson,
Secretary

National Bureau of Standards
Ernest Ambler, Acting Director

National Bureau of Standards Certificate

Standard Reference Materials

Tris(hydroxymethyl)aminomethane (922)
Tris(hydroxymethyl)aminomethane hydrochloride (923)

B. R. Staples

These Standard Reference Materials are intended for use in preparing secondary standard pH buffer solutions in the physiological range. Such buffers are used in the clinical laboratory in cases where phosphate buffers would cause undesirable side reactions or do not adequately approximate the required variation of pH with temperature. Coulometric assay indicates the tris(hydroxymethyl)aminomethane to be 99.99 ± 0.02 mole percent after drying in a vacuum oven at 70 °C for 24 hours. Coulometric assay of the tris(hydroxymethyl)-aminomethane hydrochloride as received was 99.69 ± 0.05 mole percent.

The pH(S) values listed below correspond to $\log(1/a_H)$, where a_H is a *conventional* activity of the hydrogen (hydronium) ion referred to the standard state on the scale of molality. The values were derived from the emf of cells without liquid junction by the method of calculation described by Bates [1]. The uncertainty of the assigned values of pH(S) is estimated not to exceed ±0.005 unit. The values listed below apply only to the material herein certified.

The solution recommended for the calibration of pH equipment is 0.01667 molal with respect to tris(hydroxy-methyl)aminomethane and 0.0500 molal with respect to tris(hydroxymethyl)aminomethane hydrochloride. The pH(S) of this solution as a function of temperature is as follows:

°C	pH(S)	°C	pH(S)	°C	pH(S)
0	8.471	20	7.840	37	7.382
5	8.303	25	7.699	40	7.307
10	8.142	30	7.563	45	7.186
15	7.988	35	7.433	50	7.070

The name "tris(hydroxymethyl)aminomethane" is used preferentially in this certificate since it is the name commonly used in clinical laboratories. Under the system of nomenclature recommended by the International Union of Pure and Applied Chemistry this material is named 2-amino-2-(hydroxymethyl)-1,3-propanediol. It is also known by several trivial and proprietary names, e.g., "tris," "THAM," "trizma," "tromethamine," "tromethane," etc. However, the certified values given herein apply only to the material supplied as an NBS-SRM.

The tris(hydroxymethyl)aminomethane and tris(hydroxymethyl)aminomethane hydrochloride were obtained from the Sigma Chemical Company of St. Louis, Missouri.

The overall direction and coordination of technical measurements leading to the certification was performed under the chairmanship of R. A. Durst.

The technical and support aspects involved in the preparation, certification, and issuance of these Standard Reference Materials were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D.C. 20234
June 23, 1971
Revised December 13, 1973
Revised August 20, 1976

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

These Standard Reference Materials (SRM's) are intended for "in vitro" diagnostic use only.

This material is for use in preparing standard buffer solutions for use in clinical chemistry. A buffer solution having the pH values certified on the face of this certificate is 0.01667 molal with respect to tris(hydroxymethyl)-aminomethane and 0.0500 molal with respect to tris(hydroxymethyl)aminomethane hydrochloride. The preparation of this solution is described in the following paragraph.

Transfer 7.800 g of tris(hydroxymethyl)aminomethane hydrochloride (SRM 923) and 1.999 g of tris(hydroxymethyl)aminomethane (SRM 922) to a 1-litre volumetric flask. (The figures given are weights in air; bouyancy corrections are not required.) Dissolve and fill to the mark with distilled water at 25 °C. The distilled water should not contain dissolved carbon dioxide and should have a conductivity no greater than 2×10^{-6} $S{\cdot}cm^{-1}$ (2×10^{-6} $ohm^{-1} cm^{-1}$). Carbon dioxide-free water can be prepared by boiling a good grade of distilled water for 10 minutes and guarding it with a tube of carbon dioxide absorbant. The certified pH(S) values apply for materials as received. No particular drying procedures are necessary. The tris(hydroxymethyl)aminomethane should not be heated above 80 °C and as a precaution the tris(hydroxymethyl)aminomethane hydrochloride should not be exposed to temperatures above 40 °C.

CAUTION: Reference electrodes containing linen-fiber or dialysis-membrane junctions should not be used with this buffer. Such junctions produce large liquid-junction potentials, drift, and long equilibrium times. Reference electrodes with other types of liquid junctions may also exhibit operational pH values which differ from the assigned pH(S) by up to 0.05 unit. For this reason, solutions of this buffer are not recommended as primary standards of pH except in cases where a liquid junction is not involved. This buffer is useful, however, as a secondary standard in cases where the phosphate buffers are not satisfactory, as noted above.

Standard Reference Material 922 should be stored in a well-closed container at room temperature. Exposure to high temperatures (above 50 °C) and direct sunlight should be avoided. Experience has shown this compound to be stable in storage for at least 12 years [2]. If the material degrades such that the certified values are changed, the users will be notified by NBS. It is recommended that this material not be used after 5 years from the date of purchase.

Standard Reference Material 923 should be stored in a well-closed container at room temperature, preferably in a desiccator. Exposure to high temperatures (above 40 °C) and direct sunlight should be avoided. SRM 923, tris(hydroxymethyl)aminomethane hydrochloride, is quite hygroscopic even at relative humidities of 30 to 50 percent [3]. Experience at NBS has indicated that, properly stored, this material is stable for at least 5 years. Weighing and other manipulations of SRM 923 should be avoided under conditions when the relative humidity exceeds 50 percent. If the material degrades such that the certified values are changed, the users will be notified by NBS. It is recommended that this material not be used after 5 years from the date of purchase.

This buffer solution is sensitive to contamination by carbon dioxide [4]. If the solution is to maintain the assigned pH(S) for a few weeks the exclusion of carbon dioxide is essential. The solution should be replaced after a few weeks, or sooner if mold or sediment appear, or if it is repeatedly exposed to air containing carbon dioxide.

References:

[1] R. G. Bates, Revised Standard Values for pH Measurements From 0 to 95 °C, J. Research NBS **66A,** 179 (1962).

[2] J. A. Riddick, Amine Buffers as Acidimetric Standards, Ann. N.Y. Acad. Sci. **92,** 357 (1961).

[3] Sigma Tentative Technical Bulletin No. 106 B, Sigma Chemical Company, St. Louis, Mo., revised 1967.

[4] R. G. Bates and H. B. Hetzer, Absorption of Carbon Dioxide by Solutions of 2-amino-2-(hydroxymethyl)-1,3-propanediol, Anal. Chem. **33,** 1285 (1961).

These Standard Reference Materials have been measured and certified at the laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standards Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D. C. 20234

The date of issuance and certification of these Standard Reference Materials was June 23, 1971.

U. S. Department of Commerce
Frederick B. Dent
Secretary

National Bureau of Standards
Richard W. Roberts, Director

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 924
Lithium Carbonate

This Standard Reference Material is certified as a chemical of known purity. It is intended primarily for use in calibration and standardization of procedures employed in clinical analysis and for the routine critical evaluation of daily working standards used in these procedures.

Purity . . . . $100.0_5 \pm 0.0_2$ Percent

The purity shown is based on the determination of the carbonate ion by coulometric acidimetry. The molecular weight for lithium carbonate employed in the calculations is 73.9486. This value is based on a mass-spectrometrically determined value of 6.9696 for the atomic weight of lithium in this sample. The uncertainty shown represents the 95-percent confidence interval of the mean based on 16 determinations. The assay in excess of 100 percent may be due to anion impurities of lower molecular weight than the carbonate ion, e.g., hydroxide.

This Standard Reference Material is of limited certification because no actual determination of lithium content was made. The certification is based on the analysis of the carbonate anion and the proven absence (or presence in trace quantities) of metallic cations.

The lithium carbonate used for this Standard Reference Material was obtained from the J. T. Baker Chemical Company of Phillipsburg, New Jersey. Analyses were performed by G. Marinenko, M. Darr, E. L. Garner, T. C. Rains, and T. A. Rush.

The overall direction and coordination of technical measurements leading to certification were under the chairmanship of R. A. Durst.

The technical and support aspects concerning preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

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J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

The lithium carbonate meets or exceeds the specifications for reagent grade lithium carbonate as given in Reagent Chemicals, 4th edition, published by the American Chemical Society. A semi-quantitative survey for trace contaminants by emission spectroscopy showed no significant metallic impurities. Atomic absorption and flame emission spectrometry showed neither alkali metal nor alkaline-earth impurities in excess of 1 ppm except calcium (4 ppm).

This Standard Reference Material is intended for "in vitro" diagnostic use only.

This material is intended for use as a standard for determination of lithium in clinical chemistry. For best results using either atomic absorption spectroscopy or flame emission photometry it is necessary that lithium be determined against a background of sodium and potassium.

A "standard" solution containing 1.00 mmol of lithium per liter may be prepared as follows. Dry SRM 924 for 4 hours at 200 °C, then cool to room temperature in a desiccator. Dissolve 73.91 mg of SRM 924 in 50 ml of deionized water and 20 ml of 0.1N HCl (ACS Reagent Grade). Dilute to the mark with deionized water and mix well in a 2-liter class-A volumetric flask.

A "blank" solution containing 140 mmol of sodium per liter and 5 mmol of potassium per liter may be prepared as follows: Dissolve 8.18 g of sodium chloride (SRM 919) and 0.373 g of potassium chloride (SRM 918) in deionized water. Bring to the mark of a 1-liter flask with deionized water and mix well.

Working standards containing 0.10 or 0.20 mmol of lithium, 14 mmol of sodium and 0.5 mmol of potassium per liter may be prepared as follows. To each of two 100-ml volumetric flasks add 10 ml of "blank solution". Add exactly 10.00 ml of "standard solution" to one flask and exactly 20.00 ml of "standard solution" to the second flask using class-A volumetric pipettes. Dilute each flask to the mark with deionized water and shake well.

This Standard Reference Material should be stored in the well-closed original bottle under normal laboratory conditions.

The solutions of SRM 924 are stable indefinitely when stored in a well-stoppered, all-glass container. All such solutions should be clear and display no turbidity.

References:

[1] J. Pybus and G. N. Bowers, Jr., Serum lithium determination by atomic absorption spectroscopy, in Standard Methods of Clinical Chemistry, Vol. 6, R. P. McDonald, editor-in-chief, pp 189-192, Academic Press, Inc., New York, N. Y. (1970).

[2] A. L. Levy and E. M. Katz, A comparison of serum lithium determinations using flame photometry and atomic absorption. Clin. Chem. **15**, 787 (1969).

This Standard Reference Material has been measured and certified at the laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D. C. 20234

The date of issuance and certification of this Standard Reference Material was February 23, 1972.

U. S. Department of Commerce
Frederick B. Dent
Secretary

National Bureau of Standards
Richard W. Roberts, Director

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 925
4-Hydroxy-3-methoxy-DL-mandelic Acid (VMA)

A. J. Fatiadi and R. Schaffer

This Standard Reference Material is certified as a chemical of known purity. It is intended primarily for use in the calibration and standardization of procedures employed in clinical analysis and for routine critical evaluation of the daily working standards used in these procedures.

Purity	99.4	percent
Uncharacterized Compound	maximum 0.5	percent
Keto-VMA	maximum 0.1	percent
Volatile Matter	0.02	percent
Ash	0.004	percent

The value of purity has an estimated inaccuracy of 0.4 percent. The VMA for this Standard Reference Material was prepared at the National Bureau of Standards. Analyses were performed by A. Cohen, B. Coxon, E. R. Deardorff, R. A. Durst, D. P. Enagonio, A. J. Fatiadi, J. E. Fearn, B. Greifer, G. Marinenko, W. P. Schmidt, and J. K. Taylor of the Analytical Chemistry Division.

The overall direction and coordination of technical measurements leading to certification were under the chairmanship of R. Schaffer.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D. C. 20234
May 24, 1973
Revised December 26, 1973

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

The Standard Reference Material, dried to constant weight in a vacuum desiccator over concentrated sulfuric acid, showed little tendency to gain weight when exposed to laboratory air for 24 h. Losses in weight on heating for 1 h at 56 °C at a vacuum of 0.1 mm Hg (13 Pa) averaged 0.017 percent, with little additional loss on further heating (1 h). Prolonged heating (8 h) caused gradual decomposition.

The ash content was measured using 1.0-g samples.

Solutions of VMA exposed to light and air are unstable; originally colorless solutions turn pale yellow on standing. The initial photo-oxidation product is keto-VMA (4-hydroxy-3-methoxybenzeneglyoxylic acid). A procedure for the preparation of the previously unreported keto-VMA will be published. In acetone the rate of decomposition of VMA was found by spectrophotometry at 335 nm to be about 1 percent per day. Solutions of VMA in water, ethyl acetate, or 0.01 mol/liter hydrochloric acid did not change appreciably (< 0.5 percent) during storage for three days in the dark at room temperature, or when refrigerated for three months at 3-5 °C. Instability of VMA in solutions exposed to air was found to be in the order acetone > water > ethanol> methanol > ethyl acetate.

Analysis of the Standard Reference Material (SRM) by thin-layer chromatography (tlc) revealed two impurities. When 0.5mg of this SRM was applied as a spot on non-activated silica gel G plates, developed with 4:4:1 (v:v:v) chloroform-acetic acid-water (solvent A) followed by spraying with 3 mol/liter sulfuric acid in methanol and heated for 2 min. in an oven at 110 °C showed VMA at R_F 0.63 and keto-VMA at R_F 0.92. The amount of keto-VMA detected was equivalent to 0.5 - 1.0 μg of the pure keto-VMA used as a standard. However, at least some of the keto-VMA is a chromatographic artifact. Two-dimensional tlc revealed keto-VMA in two locations, originating respectively from the positions that the VMA and the keto-VMA had occupied after the initial direction of chromatography. Furthermore, tlc of 0.1-mg quantities of keto-VMA with solvent A, followed by sulfuric acid charring, revealed the presence of a small proportion of vanillin, possibly as an artifact due to the instability of the keto-VMA in this chromatography. The second impurity, which is as yet uncharacterized, was revealed at R_F 0.19 by tlc of the SRM on silica gel GF with solvent A followed by exposure to ammonia vapor and irradiation at 366 nm. (VMA and keto-VMA are also detected by this technique.) With the silica gel GF and use of 1:1 (v:v) benzene-ethyl acetate or 5:4 (v:v) ethyl acetate-cyclohexane, the two impurities and the VMA were detected at R_F values similar to those given with solvent A. An attempt was made to isolate the uncharacterized impurity from 50 mg of SRM 925 by means of preparative thin-layer chromatography. However, this impurity underwent decomposition after being extracted and concentrated and was not detectable on chromatography by the previously employed technique. A definite proportion of the uncharacterized impurity in the SRM could not be assigned, but it is assumed to be less than 0.5 percent.

Phase solubility analyses of the SRM with 1:1 (v:v) benzene-ethyl acetate and with 5:4 (v:v) ethyl acetate-cyclohexane were performed by shaking mixtures for 8 days followed by gravimetric analysis of the solutions. The purity of the VMA appeared to be in the range 99.6-99.9 percent; however, tlc showed that both impurities remained in the solid phase. Thus, the results of these phase solubility analyses were not meaningful. In part this may be a consequence of the aforementioned instability of VMA.

Samples of VMA were titrated with standardized sodium hydroxide using a pH meter to the first inflection point of the titration curve. Complete titrations also were made in which both the carboxyl and phenolic groups were titrated. The pH values at the 0.5 and 1.5 equivalent points were 3.75 and 9.80, respectively, corresponding to the pK_1 and pK_2 for the compound. At the pH value 6.80, corresponding to the first inflection point, it was calculated that 99.91 percent of the carboxyl group is titrated; accordingly, the acid equivalent measured was increased by 0.09 percent. The sample contains a carboxylic acid equivalent of 99.83 percent.

The melting range (decomposition) of this SRM was 133-134 °C when measured in an open capillary tube heated at 1 °C per min.

Elemental analysis. Calculated for $C_9H_{10}O_5$ (198.17); C = 54.54; H = 5.09; O = 40.37. Found: C = 54.6; H = 5.1; O = 40.3.

This Standard Reference Material was prepared at the National Bureau of Standards by slowly adding glyoxylic acid to an ice-cold alkaline solution of guaiacol, with mechanical stirring. The product was purified by successive recrystallizations from ethyl acetate-cyclohexane and 2-butanone-cyclohexane mixtures. (Details of this one-step synthesis of VMA will be published.)

The synthetic material was further characterized by gas-liquid chromatography (GLC) after per(trimethylsilylation) with TRI-SIL-Z and showed R_t 17.2 min. using a glass column (1.8 m x 2 mm i.d.)of Chromosorb Q (60-80 mesh) with 3 percent OV-17 as liquid phase, isothermal at 150 °C; injection port at 180 °C; flame-ionization detector at 200 °C; nitrogen flow-rate at 25 ml per min.; samples size 10 μg in acetone (1 μg/μl). Infrared spectrum maxima (KBr pellet): 1870, 1748 and 1715 cm^{-1} (C=O stretch). Nmr (methyl sulfoxide-d_6): singlet at δ 3.80 ($-OCH_3$), multiplet at δ 6.8 (aromatic protons), broad peak at δ 8.73 (-COOH). Ultraviolet spectrum: λ_{max} in 1.0 mol/liter K_2CO_3 (10-15 min. after dissolution) at 251 nm (ϵ_M 1.022), 293 nm (0.429), and at 347 nm (0.001).

> Note: The use of proprietary designations in this certificate are for information only, and should not be construed as an endorsement of the product by either the Department of Commerce or the National Bureau of Standards.

This Standard Reference Material is for "in vitro" diagnostic use only.

This material is for use as a standard in the determination of VMA (3-methoxy-4-hydroxymandelic acid) in clinical chemistry. A "stock" standard solution containing 1 mg/ml of VMA may be prepared as follows. Accurately weigh 100 mg of SRM 925; transfer to a 100-ml volumetric flask and dissolve in 0.01N hydrochloric acid. Dilute the contents to the mark, and transfer to a glass-stoppered, brown bottle. This solution should be stored in a refrigerator at 4 °C.

To prepare VMA solutions of lower concentration, the appropriate aliquot is pipetted into a 100-ml volumetric flask and diluted to the mark with 0.01N hydrochloric acid. These diluted "working" standard solutions should be kept in a glass-stoppered, brown bottle at 4 °C and discarded after one week [1,2].

This Standard Reference Material should be kept in the well-closed, original bottle and stored in a desiccator. Refrigeration at 4 °C, or preferably −20 °C, is recommended. The SRM and desiccator should be allowed to warm to room temperature before opening. The SRM should not be subjected to direct sunlight or to artificial ultraviolet sources. Standard Reference Material 925 is being kept under close surveillance at NBS and if the material degrades beyond the limits certified, purchasers will be notified by NBS. It is recommended that the material not be used after 5 years from the date of purchase.

The "stock" standard solution containing 1 mg/ml, prepared as described above is stable for about 3 months when stored in a glass-stoppered, brown bottle in a refrigerator at 4 °C. The dilute "working" standard solution should be prepared weekly [1,2].

All constituted solutions of VMA should be clear and show no indication of turbidity.

References:

[1] Sunderman, F. W., Jr., Colorimetric determination of VMA in urine, in Standard Methods of Clinical Chemistry, Vol. 6, R. P. McDonald, editor-in-chief, Academic Press, Inc., New York, N. Y., 1970, pp 99-106.

[2] Tietz, N. W., Fundamentals of Clinical Chemistry, W. B. Saunders Co., Philadelphia, Pa. 1970, pp 577-580.

This Standard Reference Material has been measured and certified at the laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D. C. 20234

The date of issuance and certification of this Standard Reference Material was May 24, 1973.

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Acting Director

National Bureau of Standards Certificate

Standard Reference Material 926

Bovine Serum Albumin
(Total Protein Standard)

D. J. Reeder and R. Schaffer

This Standard Reference Material (SRM) is certified as a chemical of known purity. It conforms to the specifications for bovine serum albumin (lyophilized powder) approved by the National Committee for Clinical Laboratory Standards [1]. A 7-percent solution of this albumin is issued as a standardized solution (SRM 927) for use in the calibration and standardization of procedures for total serum protein determinations and for routine critical evaluation of daily working standards used in these procedures.

Table 1. Certified Data

Constituent	Mass Fraction Found
Ash	0.0006 ±0.0001
Carbohydrate	
Total hexose	0.00034 ±0.00003
Sum of citrate, pyruvate, lactate	0.00015 ±0.00002
Lipid (Total)	0.0003 ±0.0003
Non-protein amino compound, as norleucine	0.00002 ±0.00002
Water	
Karl Fischer method	0.048 ±0.002
Vacuum oven method	0.0475 ±0.0006
Protein impurities (Dimer)	
Cellulose acetate	None detected
Gel electrophoresis (polyacrylamide gel)	0.043 ±0.002
Gel chromatography (Sephadex)	0.050 ±0.003
Spectral properties	**Absorbance**
Ultraviolet, (A_{252}/A_{279} ratio)	0.485 ±0.009
Soret band, (A_{405} at 70 g/L)	0.182 ±0.002
Visual, (A_{500} at 70 g/L)	0.048 ±0.002
(A_{600} at 70 g/L)	0.023 ±0.002

The uncertainty is expressed as one standard deviation. The spelling "liter" and its symbol "L" are recommended usage in the United States (See Federal Register, Vol. 41, 54018, Dec. 10, 1976).

The bovine serum albumin used for this Standard Reference Material was prepared by the Research Division, Miles Laboratories, Inc., Kankakee, Illinois. Analyses were performed by R. G. Christensen, A. Cohen, R. Deardorff, R. Durst, H. Hertz, B. Howell, S. Margolis, J. Maurey, D. J. Reeder, L. Sniegoski, P. Verdier, H. L. Wagner, F. W. Wang, and W. Yap.

The overall direction and coordination of technical measurements leading to the certification were under the chairmanship of R. Schaffer.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D.C. 20234
March 10, 1977
Revised July 18, 1977

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

The analytical techniques used in the certification of this Standard Reference Material are generally those recommended in NCCLS Approved Standard: ACC-1 [1]. Non-protein amino compounds were determined by fluorescence methodology [2].

Supplementary information for this Standard Reference Material is given in Table 2; the amino acid composition of the Bovine Serum Albumin in Table 3; and the fatty acid composition of the included lipid in Table 4. These data are not certified, but given for information only.

Table 2 — Supplementary information on SRM 926, Bovine Serum Albumin

Refractive index increment (dn/dc) (cm^3/g)	0.18440 ±0.00025
Partial specific volume (in vacuo) (cm^3/g)	0.7332 ±0.0007
Nitrogen analysis (Kjeldahl) (mg/g)	163.98 ±1.15
Immunoelectrophoresis	No protein impurities detected
Molecular weight (g/mol)[a]	68,400 ±110
Number-average molecular weight of 7-percent solution, M_n/C_o^b	$9.73 \times 10^5 \pm 0.11 \times 10^5$

[a]Weight-average molecular weight, M_w, determined by equilibrium centrifugation.

[b]Determined by membrane osmometry, where C_o is concentration of albumin standard (SRM 927).

Table 3 — Amino acid composition of SRM 926, Bovine Serum Albumin

Amino Acid	Residues per 100 Residues
Aspartic acid	9.13
Threonine	5.49
Serine	4.61
Glutamic acid	13.89
Proline	5.73
Glycine	2.72
Alanine	7.98
1/2-Cystine	5.51
Valine	6.30
Methionine	0.67
Isoleucine	1.97
Leucine	11.03
Tyrosine	3.36
Phenylalanine	4.86
Lysine	9.98
Histidine	2.85
Arginine	3.93
Tryptophan	Not determined

Table 4 — Fatty acid composition by gas chromatography-mass spectrometry of the extracted lipid.

Compound	Relative Amount
Methyl benzoate	4.4
Methyl decanoate	3.0
Methyl dodecanoate	3.9
Methyl tetradecanoate (myristate)	10
Methyl pentadecanoate	6.9
A phthalate ester	7.1
Methyl hexadecenoate (a)	20
Methyl hexadecanoate (palmitate)	45
Dibutyl phthalate	100
Methyl oleate	12
Methyl octadecanoate (stearate)	23
Methyl eicosanoate	8.1

a = probable identity

This Standard Reference Material is intended for "in vitro" diagnostic use only.

This Standard Reference Material should be stored in a well-closed container at a temperature not to exceed 4 °C. Storage in a desiccator is recommended. It should not be subjected to sunlight or ultraviolet radiation. Under proper storage, this SRM is expected to be stable for at least 3 years. Samples of this material will be monitored and should the certified properties degrade beyond the limits specified, purchasers will be notified by NBS. It is recommended that material not be used after 3 years from date of purchase.

The lyophilized Standard Reference Material is extremely hygroscopic and will generally increase in moisture content under most weighing conditions. It is recommended that, for applications requiring only portions of the material, the container should be opened and samples weighed under controlled atmospheric conditions, e.g., a dry nitrogen atmosphere in a glove box or similar type of isolation chamber.

Albumin solutions may be prepared from the lyophilized albumin powder by adding the powder to the surface of the liquid and allowing the powder to dissolve by diffusion into the liquid. Avoid disturbing the solution during this process. If dry powder is present when the solution is mixed,the clumps that formed will be found sparingly soluble and may take several days to dissolve.

The SRM should not be used if the material takes on a color other than the normal white appearance. Another sign of degradation of the albumin is insolubility of the powder when preparing solutions at a concentration of 1% or less.

References:

[1] Standardized ProteinSolution (BovineSerumAlbumin), Approved Standard: ACC-1, National Committee for Clinical Laboratory Standards, Villanova, Pa., 1972.

[2] Reeder, D.J., Sniegoski, L.T., and Schaffer, R., o-Phthalaldehyde for the Fluorimetric Assay of Non-protein Amino Compounds, submitted for publication to Clinical Chemistry.

This Standard Reference Material has been measured and certified at the Laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquries should be addressed to:

Office of Standard Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D.C. 20234

The date of issuance and certification of this Standard Reference Material was March 10, 1977.

NOTE: The use of proprietary names are for identification purposes only and do not constitute NBS endorsement of any particular product.

U.S. Department of Commerce
Juanita M. Kreps
Secretary
National Bureau of Standards
Ernest Ambler, Acting Director

National Bureau of Standards Certificate

Standard Reference Material 927

Bovine Serum Albumin (7% Solution) (Total Protein Standard)

Dennis J. Reeder and Robert Schaffer

This Standard Reference Material (SRM) is certified as a solution of known protein concentration and purity. It conforms to the specification for standardized protein solution approved by the National Committee for Clinical Laboratory Standards (NCCLS) [1]. SRM 927 was prepared from the same lot of bovine serum albumin as SRM 926, brought to proper ionic strength with sodium chloride and to proper pH with sodium hydroxide. It was sterilized by membrane filtration. This SRM is intended primarily for use in the calibration and standardization of procedures employed in clinical analyses for total serum protein determinations and for routine critical evaluation of daily working standards used in these procedures.

Table 1. Certified Data

Peptide mass (g/L)*	
Biuret method [2]	70.45 ± 0.10
Kjeldahl method	70.48 ± 0.50
Method using optical density at 278 nm	70.77 ± 0.23
pH	6.66 ± 0.01
Sodium ion, substance concentration (mol/L)	0.0291 ± 0.0004
Chloride ion, substance concentration (mol/L)	0.0210 ± 0.0002
Volume (mL)	2.06 - 2.28

*The spelling of "liter" and its symbol "L" are recommended usage in the United States (Federal Register, **41**, 51089, December 10, 1976).

The inaccuracies expressed in the above table are one standard deviation (σ), except for the peptide mass by biuret method. In the analysis of the biuret data, regression curves were developed for three separate experiments involving at least 80 separate correlated measurements. Thus, the final data is expressed in terms of standard error ($\sigma/\sqrt{n}$), rather than standard deviation.

The bovine serum albumin solution was prepared by the Research Division, Miles Laboratories, Inc., Kankakee, Illinois. Analyses were performed by R. G. Christensen, A. Cohen, R. Deardorff, R. Durst, H. Hertz, B. Howell, S. Margolis, J. Maurey, D. Reeder, L. Sniegoski, W. Yap, P. Verdier, and H. L. Wagner.

The overall direction and coordination of technical measurements leading to the certification were under the chairmanship of R. Schaffer.

Washington, D.C. 20234
December 31, 1979
(Revision of Certificates dated 3-16-77 and 7-18-77)

George A. Uriano, Chief
Office of Standard Reference Materials

(over)

This Standard Reference Material is intended for "in vitro" diagnostic use only.

This material is for use as a standard in clinical chemistry. It is intended for use primarily as a reference standard for assays of total protein by colorimetric methods. A biuret method [2], modified to a 60-minute incubation, was employed to assay the comparison solutions prepared from SRM 926, and the standardized ampouled solution. It is suggested that this biuret method be used to standardize laboratory-prepared protein solutions and "normal" serum pools. Such standardized "normal" sera could then be used to calibrate refractometers or other instruments for serum protein estimations. Standard Reference Material 927 may be used for other procedures, such as gel diffusion, amino acid analysis, electrophoresis, nitrogen assays, or other tests which require well-characterized protein.

WARNING

This Standard Reference Material is not to be used in standardizing dye-binding tests, for directly checking precalibrated refractometers, or as an immunochemical standard. It is not recommended for use in billirubin standardization.

Protein solutions of lower concentration may be prepared by transfer of the appropriate aliquot to a 25-mL volumetric flask and diluted. Diluents are not furnished with the Standard Reference Material. An aqueous diluent, such as 0.15 mol/L sodium chloride solution, may be used.

This Standard Reference Material is supplied to the user in sealed ampoules. It must be stored such that the temperature of the solution does not exceed 4 °C; nor may the solution be allowed to freeze. Once an ampoule is opened, the solution should be used promptly. Storage of opened ampoules is not recommended. Under proper storage in sealed ampoules this SRM is expected to be stable for 3 years. Stocks of this SRM kept at NBS will be monitored and if degradation is evident, purchasers will be notified. It is recommended that this material not be used after 3 years from date of purchase.

References:

[1] Standardized Protein Solution (Bovine Serum Albumin), Approved Standard: ACC-1, National Committee for Clinical Laboratory Standards, Villanova, Pa., 1972.

[2] B. T. Doumas, Standards for Total Serum Protein Assays - A Collaborative Study. Clin. Chem. **21** (8), 1159-1166 (1975).

[3] United States Pharmacopeia, 18th Revision, p. 855, Class A, United States Pharmacopeial Convention, Rockville, Md. 1970 (see also 19th Revision, p. 712, 1975).

This Standard Reference Material has been measured and certified at the laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D.C. 20234

The date of issuance and certification of this Standard Reference Material was March 16, 1977.

The sterile ampouled solution used for this Standard Reference Material was prepared from the same lot of bovine serum albumin as SRM 926. The following properties, which equal or exceed those required by NCCLS Approved Standard ACC-1, were measured on aqueous solutions of SRM 926. The uncertainties are expressed as one standard deviation.

Constituent	Mass Fraction Found
Ash	0.0006 ± 0.0001
Carbohydrate	
Total hexose	0.00034 ± 0.00003
Sum of citrate, pyruvate, and lactate	0.00015 ± 0.00002
Lipid (total)	0.0003 ± 0.0003
Non-protein amino compound, as norleucine	0.00002 ± 0.00002
Water	
Karl Fischer method	0.048 ± 0.002
Vacuum oven method	0.0475 ± 0.0006
Protein impurities (Dimer)	
Cellulose acetate	None detected
Gel electrophoresis	0.043 ± 0.002
Gel chromatography	0.050 ± 0.003

Spectral properties	Absorbance
Ultraviolet (A_{252}/A_{279} ratio at 70 g/L)	0.485 ± 0.009
Soret band (A_{405} at 70 g/L)	0.182 ± 0.002
Visual (A_{500} at 70 g/L)	0.048 ± 0.002
(A_{600} at 70 g/L)	0.023 ± 0.002

The certified protein concentration is based on the mass of polypeptide material dissolved per liter of solution, where peptide material is defined as albumin powder less its content of ash, carbohydrate, lipid, non-protein amino compounds, and water.

Supplementary information on SRM 926, Bovine Serum Albumin	
Refractive index increment (dn/dc) (cm^3/g)	0.18440 ± 0.00025
Partial specific volume (in vacuo) (cm^3/g)	0.7332 ± 0.0007
Nitrogen analysis (Kjeldahl) (mg/g)	163.98 ± 1.15
Immunoelectrophoresis	No protein impurities detected
Molecular weight[a]	68,400 ± 110
Number-average molecular weight[b] of albumin (as 7-percent solution)	68,500 ± 780

[a]Weight-average molecular weight, Mw, determined by equilibrium centrifugation.

[b]Value determined by membrane osmometry which gave $Mn/Co = 9.73 \times 10^5 \pm 0.11 \times 10^5$ mL/mol, where Mn is the number-average molecular weight and Co is the concentration of albumin standard (SRM 927).

U.S. Department of Commerce
Elliot L. Richardson,
Secretary

National Bureau of Standards
Ernest Ambler, Acting Director

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 928
Lead Nitrate
(Clinical Standard)

This Standard Reference Material is certified for use as an assay standard for lead. It is intended primarily for use in the calibration and standardization of procedures employed in clinical analysis and for the routine critical evaluation of daily working standards used in these procedures.

Assay . . . . 100.00 ± 0.03 percent

The assay shown is based on the determination of lead in the material as received; drying being unnecessary. Lead is precipitated as the chromate using a slight excess of potassium dichromate (SRM 136c). The lead chromate is removed by filtration and the excess chromate ion determined spectrophotometrically. Details of this method are reported elsewhere [1]. The molecular weight of lead nitrate employed in the calculation is 331.219. This value is based on a mass-spectrometrically determined value of 207.209 for the atomic weight of lead in this sample. The uncertainty shown represents two standard deviations of a single measurement based on 16 determinations with allowances for known sources of possible error.

A semi-quantitative survey for trace metals by emission spectroscopy indicated the following: silver, 2 μg/g; chromium, 3 μg/g; nickel, 3 μg/g. No other metals were detected.

The lead nitrate used for this Standard Reference Material was obtained from the J. T. Baker Chemical Co., Phillipsburg, New Jersey. Chemical analyses were performed by T. J. Murphy and J. W. Gramlich. Spectroscopic analyses were performed by C. S. Annell and D. Golightly of the United States Geological Survey.

The overall direction and coordination of technical measurements leading to certification were under the chairmanship of I. L. Barnes.

The technical and support aspects concerning preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D.C. 20234
February 27, 1976
Revised May 19, 1976

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

This material was examined for compliance with the specifications for reagent grade lead nitrate as given in Reagent Chemicals, 5th edition, published by the American Chemical Society. The material met or exceeded the requirements in every respect.

This Standard Reference Material is intended for "in vitro" diagnostic use only.

This material is intended for use as a standard for lead determination in clinical chemistry.

Because of the instability of non-acidified aqueous lead solutions at the working levels it is recommended [2] that three levels of concentration be used.

(a) A stock standard solution containing 50 mmol/l is prepared by dissolving 1.6561 g of SRM 928 in ion-free water. If the solution is cloudy or a precipitate forms, add a few drops of ammonium hydroxide. Mix, dilute to 100 ml in a calibrated volumetric flask and transfer immediately to a previously acid-washed, water-rinsed, dry polyethylene bottle. This solution is stable for six months. [2].

The above directions are directly quoted from the reference given. Users are cautioned that carbonate-free ammonium hydroxide not in excess of 0.2 ml should be added. Otherwise, insoluble basic lead salts will form. However, experience at NBS indicates that this material will easily dissolve *without cloudiness* when high-purity water is used.

(b) An intermediate solution containing 500 μmol/l is prepared by a 1:100 dilution of the above stock solution. This solution may be stored in a capped polyethylene bottle at room temperature for one month [2].

(c) Working standard solutions of 0.5, 1.0, 2.5, and 5.0 μmoles/l should be prepared each time an analysis is performed [2].

Note: Dilute aqueous lead standards remain stable for less than three hours. Lead is readily adsorbed on the surfaces of glass and plastic containers and this reaction is accelerated by exposure to light, particularly ultraviolet light [3]. It is recommended that very dilute aqueous lead solutions be prepared in a darkened room and protected from light [4].

This Standard Reference Material should be stored in the tightly-closed original bottle under normal laboratory conditions. Tests show the material to be dry as received and not to adsorb appreciable water when exposed to a 90 percent relative humidity atmosphere for 5 days.

The solutions of SRM 928 are stable as described above. At time of use these solutions should be clear and display no turbidity.

References:

[1] E. J. Catanzaro, T. J. Murphy, W. R. Shields and E. L. Garner, J. Res. NBS 72A, 261-267 (1968).

[2] L. Kopito and H. Shwachman, Measurement of lead in blood, urine, and hair by atomic absorption spectroscopy, Standard Methods of Clinical Chemistry, Vol. 7, G. R. Cooper, editor-in-chief, pp. 151-162, Academic Press, Inc., New York, N.Y. 1972.

[3] L. Kopito and H. Shwachman, J. Lab. Clin. Med. 70, 326-332 (1967).

[4] Fundamentals of Clinical Chemistry, N. W. Tietz, editor, pp. 852-857, W. B. Saunders Co., Philadelphia, Pa. (1970).

The date of issuance and certification of this Standard Reference Material was June 15, 1975.

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards
Certificate of Analysis

Standard Reference Material 929

Magnesium Gluconate Dihydrate

(Clinical Standard for Magnesium)

This Standard Reference Material is certified for use as an assay standard for magnesium. It is intended primarily for use in the calibration and standardization of procedures employed in clinical analysis and for the routine critical evaluation of daily working standards used in these procedures. The material is highly purified magnesium gluconate dihydrate, $Mg\,(C_6H_{11}O_7)_2{\cdot}2H_2O$.

MAGNESIUM, weight percent: 5.403 ± 0.022

This certified value is based on a minimum sample of 25 mg of the material dried to constant weight for at least 24 hours over anhydrous magnesium perchlorate. The certified value is based on the determination of magnesium in the *dried material* by thermal ionization isotope dilution mass spectrometry. Details of this method are reported elsewhere [1].

For mass spectrometric analysis, two different methods of magnesium separation were used. The uncertainty; 0.022, is the sum of: 0.014, the possible systematic differences between the methods; and 0.008, the 95 percent confidence limit for the mean.

The magnesium gluconate dihydrate used for this Standard Reference Material was obtained from the J.T. Baker Chemical Co., Phillipsburg, N.J.

The analyses were performed by L.A. Machlan, J.R. Moody, and L.J. Moore of the NBS Inorganic Analytical Research Division.

The statistical evaluation of the data was performed by H.H. Ku, NBS Statistical Engineering Division.

The overall direction and coordination of the technical measurements leading to certification were under the chairmanship of I.L. Barnes.

The technical and support aspects concerning preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.W. Seward.

Washington, D.C. 20234
April 27, 1979

George A. Uriano, Acting Chief
Office of Standard Reference Materials

(over)

The water of hydration was determined on the dried material (see above) by the Karl-Fischer method using dry methanol and dimethyl sulfoxide (80:20) solvent. The water content was found to be 7.99 weight percent, which is in agreement with the theoretical value of 2 moles of water per mole of salt, 8.00 weight percent. Determination of water content was performed by S.A. Margolis, NBS Organic Analytical Research Division.

This Standard Reference Material is intended for "in vitro" diagnostic use only.

This material is intended for use as a standard for magnesium determination in clinical chemistry.

STORAGE: Standard Reference Material 929 should be stored in the tightly-closed, original bottle under normal laboratory conditions. Tests show this material to be hygroscopic and must be dried as directed before use; such drying will not remove water of hydration. Stored under these conditions, this material will show no significant change in properties. NBS will keep this lot under surveillance for five years after the date of last sale. If degradation occurs beyond the limit certified, NBS will notify purchasers. In the absence of such notification, the user should not use the material beyond five years after the purchase date.

A standard solution containing 5.00 mmol/L of magnesium may be prepared by placing 1.125 g of dried SRM 929 in a 500 mL volumetric flask and dissolving the material with laboratory reagent grade water.* Lower concentrations required for analysis may be prepared by accurate dilutions.

Solutions of SRM 929 prepared as above are stable for at least 60 days under normal laboratory conditions.

[1] Catanzaro, E.J.; Murphy, T.J.; Garner, E.L.; and Shields, W.R., J. Res. **NBS 70A,** No. 6, 553-558 (1966).

* Laboratory reagent grade water meeting any of the following specifications:
American Society for Testing and Materials (ASTM), D1193-Type II;
College of American Pathologists (CAP), Type II;
National Committee for Clinical Laboratory Standards (NCCLS), Type I.

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Acting Director

National Bureau of Standards Certificate

Standard Reference Material 930D

Glass Filters for Spectrophotometry

R. Mavrodineanu and J. R. Baldwin

This Standard Reference Material is intended as a reference source for the verification of the transmittance and absorbance scales of spectrophotometers. It consists of three individual glass filters in their metal holders and one empty filter holder. The holders are provided with shutters to protect the glass filters when not in use. These shutters must be removed at the time of measurements and be replaced after the measurements have been completed. Each filter bears an identification number, and the upper left corner has been removed to indicate correct placing in the metal holder.

Filter & Set Ident. Number	TRANSMITTANCE (T) Wavelength and (Bandpass) nm					TRANSMISSION DENSITY ($-\log_{10}T$) Wavelength and (Bandpass) nm				
	440.0 (2.2)	465.0 (2.7)	546.1 (6.5)	590.0 (5.4)	635.0 (6.0)	440.00 (2.2)	465.0 (2.7)	546.1 (6.5)	590.0 (5.4)	635.0 (6.0)
1-930	0.0923	0.1096	0.1023	0.0923	0.1032	1.035	0.960	0.990	1.035	0.987
2-930	0.1920	0.2161	0.2059	0.1918	0.2069	0.717	0.665	0.686	0.717	0.684
3-930	0.2929	0.3193	0.2993	0.2745	0.2777	0.533	0.4958	0.524	0.561	0.556

Date of Certification: January 1, 1981

The transmittance values (T) can be converted to percent transmittance (%T) by multiplying by 100. The transmission densities are calculated from the measured transmittance (T). These values should be indicated by the absorbance scale of the spectrophotometer if the filters are measured against air. The transmittance values given were measured against air at an ambient temperature of 23.5 °C.

The transmittance values are estimated to be accurate to within ±0.5 percent at the time of certification. This uncertainty includes the effects of the random and systematic errors of the calibration procedure, as well as possible transmittance changes of the filters during the period of calibration.

Aging of the glass may cause some filters to change transmittance by about ±1 percent over a period of approximately one year from the date of calibration. Improper storage or handling of the filters may also cause changes [5]. It is recommended that the filters in the holders be handled only by the edges with soft plastic (polyethylene) gloves and optical lens tissue. When not in use they should be stored in their holders and in the container provided for this purpose. Extended exposure to laboratory atmosphere and dusty surroundings should be avoided. In cases where verification is desirable, the filters should be returned to the National Bureau of Standards for cleaning and recalibration.

This Standard Reference Material was issued with the assistance and advice of K. D. Mielenz and J. R. DeVoe.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears and R. W. Seward.

Washington, D.C. 20234
August 1, 1977

(over)

J. Paul Cali, Chief
Office of Standard Reference Materials

The transmittance measurements were made using the high-accuracy spectrophotometer designed and built at the NBS Institute for Materials Research [1]. This instrument represents the primary transmittance standard; its transmittance accuracy was established using the double-aperture method of linearity testing [1,3,5,6]. The estimated standard deviation obtained from a set of 20 measurements of the high-accuracy spectrometer used for transmittance measurements on glass filters of the SRM 930 type is 5×10^{-5} transmittance units for transmittance between 0.1 and 0.3. The bounds of the systematic error of the instrument are estimated to be $\pm 10^{-4}$ transmittance units.

The neutral glasses for the filters were provided by Schott of Mainz, Germany, and are designated as "Jena Color and Filter Glass." The glass material was prepared by the manufacturer especially for SRM 930. It was fine-annealed and was selected for best homogeneity and minimum of inclusions and striae. The glass filters were aged at NBS for about one year prior to certification. They are of the type NG-4 and NG-5, and their nominal transmittances are 10, 20, and 30 percent [2,5]. The glasses were selected to provide a means to verify the transmittance scale of conventional spectrophotometers at three levels. The exposed surface of the glass is approximately 29 x 8 mm, measuring from a point 1.5 mm above the base of the filter holder (see figure). The empty filter holder is provided to be used in the reference beam of the spectrophotometer so that approximately equivalent conditions of stray radiations are achieved for both beams.

The transmittance of the filters depends upon the intrinsic properties of the material, wavelength, spectral bandpass, geometry of the optical beam, temperature, and positioning of the filter. A change of ambient temperature of 1 or 2 °C from 23.5 °C will not significantly affect the calibration [5]. Changes in the transmittance may be caused by surface conditions, aging of the glass, exposure to a harmful atmosphere, or careless handling as indicated on the face of this certificate [2,3,4,5]. The effective spectral bandpasses used to determine the certified values are given on the face of the certificate and the transmittance measurements are made by producting the vertical image of the slit (about 8 mm by 0.5 mm), using a convergent beam geometry with an aperture ratio f:10, in the middle of the entrance face of the glass filter. The filters were measured in the spectrophotometer in a position perpendicular to the incident light beam as shown in the attached figure.

SRM 930D is stored in a black anodized aluminum container provided with a threaded cap made of the same metal. Each filter is placed in a cylindrical cavity to prevent any contact between the filter face and the walls of the storage container. Contamination of the glass filter surface with particulate matter due to static charges is minimized through the metallic nature of the container. Each filter holder is provided with a flat leaf spring which is inserted into the cylindrical cavity to minimize damage during transportation. These springs can be removed during normal use in the laboratory.

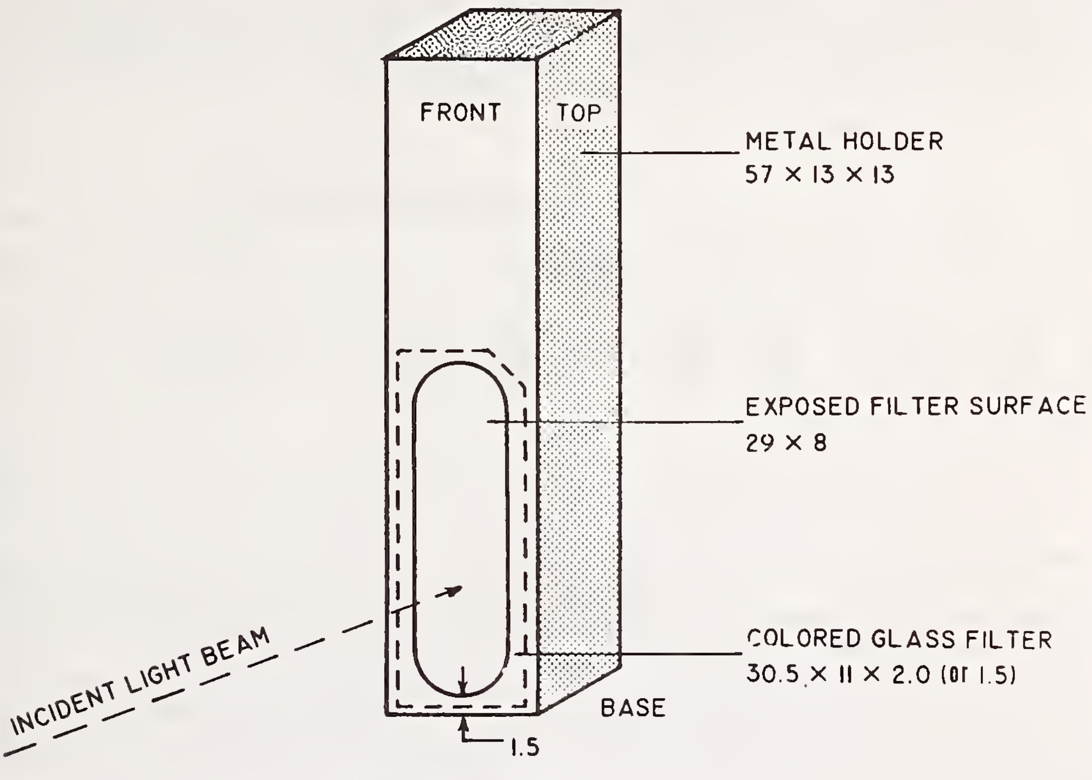
Prior to certification measurements, each filter was examined for surface defects and thorougly cleaned [5]. Should the surface of the glass filter become contaminated, no attempt should be made to clean it unless the user has the facilities to demonstrate that the cleaning treatment has not altered the glass surface or degraded the accuracy of the certified values. As the Standard Reference Material is a transfer standard, the only means available to verify its integrity is remeasuring its transmittance with a primary standard instrument similar to that used in this certification [1,4,5]. In most cases where verification, or recertification, of the transmittance values is desirable, it will be most expeditious to return the filters to the National Bureau of Standards for measurement. Prior to shipment, the NBS Office of Standard Reference Materials should be contacted regarding the conditions and cost of verification and/or recertification.

We wish to acknowledge the cooperation of George N. Bowers, Jr., M.D., of Hartford Hospital, Hartford, Connecticut; Royden N. Rand, Ph.D., of the Eastman Kodak Co. Research Laboratories, Rochester, New York; and Donald S. Young, M.D., Ph.D., of the National Institutes of Health, Bethesda, Maryland.

REFERENCES

1. R. Mavrodineanu, An Accurate Spectrophotometer for Measuring the Transmittance of Solid and Liquid Materials, NBS Journal of Research **76A**, No. 5, 405-425 (1972).
2. R. Mavrodineanu, Solid Materials to Check the Photometric Scale of Spectrophotometers, NBS Tech. Note 544, O. Menis and J. I. Shultz, Eds., pp. 6-17, U.S. Government Printing Office, Washington, D.C. 20402 (Sept. 1970); *ibid*, NBS Tech. Note 584, pp. 2-21 (December 1971).
3. K. S. Gibson, Spectrophotometry, NBS Circ. 484 (Sept. 1949).
4. Collected Papers from NBS Conference on Accuracy in Spectrophotometry and Luminescence Measurements, NBS Journal of Research **76A**, No. 5, 375-510 (1972).
5. R. Mavrodineanu and J. R. Baldwin, Glass Filters as a Standard Reference Material for Spectrophotometry-Selection, Preparation, Certification, Use, SRM 930, NBS Special Publication 260-51, U.S. Government Printing Office, Washington, D.C. 20402, 1975.
6. K. D. Mielenz and K. L. Eckerle, Spectrophotometer Linearity Testing Using the Double-Aperture Method, Appl. Optics **11**, 2294-2303 (1972).

METAL HOLDER FOR THE COLORED GLASS FILTERS*



DIMENSIONS IN mm

*Shutters not illustrated.

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Acting Director

National Bureau of Standards Certificate

Standard Reference Material 931b

Liquid Absorbance Standards for Ultraviolet and Visible Spectrophotometry

R. W. Burke and E. R. Deardorff

This Standard Reference Material is certified as solutions of known net absorbance at specific spectral wavelengths. It is intended primarily for use in the calibration and checking of accuracy of the photometric scale of narrow bandpass spectrophotometers employed in clincial analysis and for routine critical evaluation of daily working standards used in clinical spectrophotometry. This Standard Reference Material is applicable for calibrating those instruments that provide an effective spectral bandpass of 1.5 nm or less at 302 nm, 2.0 nm or less at 395 nm, 3.3 nm or less at 512 nm and 8.5 nm or less at 678 nm [1].

Filter	Net Absorbance[a] Wavelength and (Bandpass), nm			
	302(1.0)	395(1.7)	512(2.0)	678(6.5)
"I" - "Blank"	0.306 ± 0.003	0.302 ± 0.003	0.300 ± 0.003	0.115 ± 0.002
"II" - "Blank"	.607 ± .004	.605 ± .004	.602 ± .004	.230 ± .003
"III" - "Blank"	.891 ± .005	.902 ± .005	.899 ± .005	.342 ± .003

[a]Net absorbances ("I" - "Blank", "II" - "Blank", and "III" - "Blank") were determined using 10.00 mm cuvettes (SRM 932) at 25 °C. See Instructions for Use.

Absorbance measurements were performed on a high precision double-beam spectrophotometer equipped with a double monochromator. The accuracy of the photometric scale of this instrument was established with the NBS high-accuracy spectrophotometer described by Mavrodineanu [2]. The uncertainties of the certified values include all known sources of possible systematic error and the 95 percent confidence level for the mean.

While no long-term stability studies have been made on this lot (931b), studies on previous lots (931 and 931a) over a three-year period showed no degradation of the material when stored in the original sealed ampoules. Nevertheless, until additional information is forthcoming, it is recommended that this material not be used after three years from the date of purchase.

The overall direction and coordination of technical measurements leading to certification were performed under the chairmanship of I. L. Barnes.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W. P. Reed.

Washington, D.C. 20234
November 10, 1977

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

Temperature Dependence

Absorbances at various temperatures (17 to 35 °C) may be calculated using the equation

$$A_t = A_{25}[1+C_A(t-25)],$$

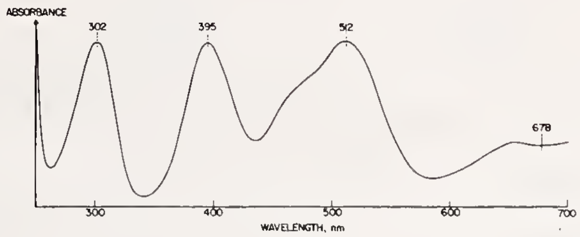
where: A_t = Absorbance at temperature t (°C)
A_{25} = Absorbance certified at 25.0 °C
C_A = Fractional change in absorbance per °C

The values of C_A, at the four wavelengths, are given below. [NOTE: At wavelength 302 nm, absorbance decreases with increasing temperature; at the other wavelengths, absorbance increases with increasing temperature.]

Wavelength, nm	C_A
302	−0.0014
395	+0.0014
512	+0.0018
678	+0.0014

Preparation of Filters

The filters were prepared by dissolving high-purity cobalt and nickel in a mixture of nitric and perchloric acids. The absorbance spectrum of the resulting solution is shown in the following figure. The maxima at 302 and 512 nm are due to absorbance by NO_3^- and $Co(H_2O)_6^{++}$, respectively. The maximum at 395 nm and the plateau at 650-700 nm are due to $Ni(H_2O)_6^{++}$. The pH of these solutions is about 1.



Warning

This Standard Reference Material is intended for "in vitro" diagnostic use only.

Instructions for Use

This material is for use as a spectrophotometric absorbance standard.

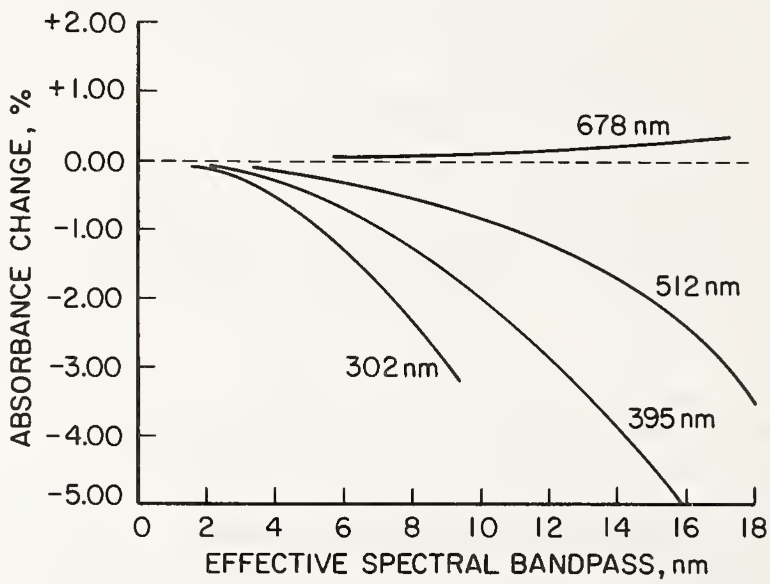
1. Select two clean 10.00 mm cuvettes free of scratches. At least one should be fitted with a ground glass or Teflon stopper to minimize evaporation. Reserve it for all sample measurements.
2. Mark each of the cuvettes to assure the same orientation in the spectrophotometer.
3. Place the cuvettes in their respective holders and fill with distilled water. (Borosilicate Pasteur-type pipettes fitted with rubber bulbs are recommended for transferring all solutions to and from the cuvettes. Soft glass pipettes, which are available commercially, contain residual amounts of ultraviolet absorbing material, but may be used after proper cleaning. Several rinses, first with isopropyl alcohol and then with distilled water, are generally adequate.)
4. Obtain the optical mismatch of the cuvettes at 302, 395, 512, and 678 nm, using the spectral bandpass limitations given on the face of the certificate.
5. Empty the cuvettes by suction without removing them from their holders, refill with distilled water and measure the absorbances again at each of the above wavelengths.

6. Repeat the emptying and refilling operation until constant absorbance readings are obtained.
7. Using the liquid filters provided, measure, in turn, the absorbance of the "Blank", "I", "II", and "III" against distilled water. Shake each ampoule before opening to remix any condensate which may have collected in the neck. (The ampoules have been prescored directly below the gold band to facilitate opening.)
8. Subtract the appropriate "Blank" reading from the absorbances obtained for "I", "II", and "III". These net absorbances should agree with the certified values within the uncertainties specified. Consult the manufacturer of the instrument if they do not.

The above instructions are for use with the standard 10-mm rectangular cuvette. For calibration of the several spectrophotometric systems used in various automated instruments, the user is referred to the instruction manual for the particular instrument.

The absorbances of these liquid absorbance standards will depend not only on the accuracy of the photometric scale, but also on the wavelength accuracy and the spectral bandpass. A mercury lamp is recommended for checking the wavelength scale. In addition, for those spectrophotometers having a hydrogen (H) or deuterium (D) source, the two emission lines at 486.1 and 656.3 nm (H) or 486.0 and 656.1 nm (D) may provide a convenient check at these wavelengths.

To insure that the measured absorbances are not significantly different from the certified values, the following restrictions are placed on the size of the spectral bandpass selected: To obtain ±0.1 percent of the true value, the effective spectral bandpass should not exceed 1.5, 2.0, 3.3, and 8.5 nm at 302, 395, 512, and 678 nm, respectively. For ±0.2 percent, the respective bandpasses should not exceed 2.2, 2.9, 4.8, and 12.3 nm. Additional information on the effect of spectral bandpass on the absorbances of these filters is given in the figure below. These curves are not to be used, however, to correct the measured absorbances.



This Standard Reference Material should be kept in the original sealed ampoules. Once opened, the material should be used immediately. No attempt should be made to reseal the ampoule. In addition, it is recommended that this Standard Reference Material not be used after three years from the purchase date.

References

[1] R. W. Burke, E. R. Deardorff, and O. Menis, J. Research, Nat. Bur. Stand. **76A**, 469-482 (1972).

[2] R. Mavrodineanu, J. Research, Nat. Bur. Stand. **76A**, 405-425 (1972).

Note: The above papers are also published in NBS Special Publication 378, Accuracy in Spectrophotometry and Luminescence Measurements, R. Mavrodineanu, J. I. Shultz, and O. Menis, editors, U.S. Government Printing Office, Washington, D.C. 20402, 1973.

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards
Certificate

Standard Reference Material 932

Quartz Cuvette for Spectrophotometry

R. Mavrodineanu and J. W. Lazar

This Standard Reference Material consists of a single, accurately calibrated cuvette that is issued for use in the production of accurate spectrophotometric data on liquids. The design and nominal dimensions of the all-quartz cuvette are shown in Figure 1. The pathlength of the cuvette is defined by the distances between the two optically transparent windows taken at several heights within the cuvette. Cuvettes issued as Standard Reference Material 932 have a nominal pathlength of 10 mm. The pathlength and parallelism are certified with an uncertainty of ±0.0005 mm as determined by measurements (at 20°C) taken at the positions indicated below.

Cuvette number   is issued with this certificate. For this cuvette the following measurements were obtained.

Height mm	Pathlength mm

The cuvette must be handled with great care and should be held only by the frosted-quartz side windows. When not in use, it should be stored in the container provided for this purpose. Extended exposure to laboratory atmosphere and dusty surroundings is to be avoided.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of O. Menis and J. A. Simpson.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D.C. 20234
August 2, 1978
(Revision of certificates
dated 11-5-73 and 6-16-77)

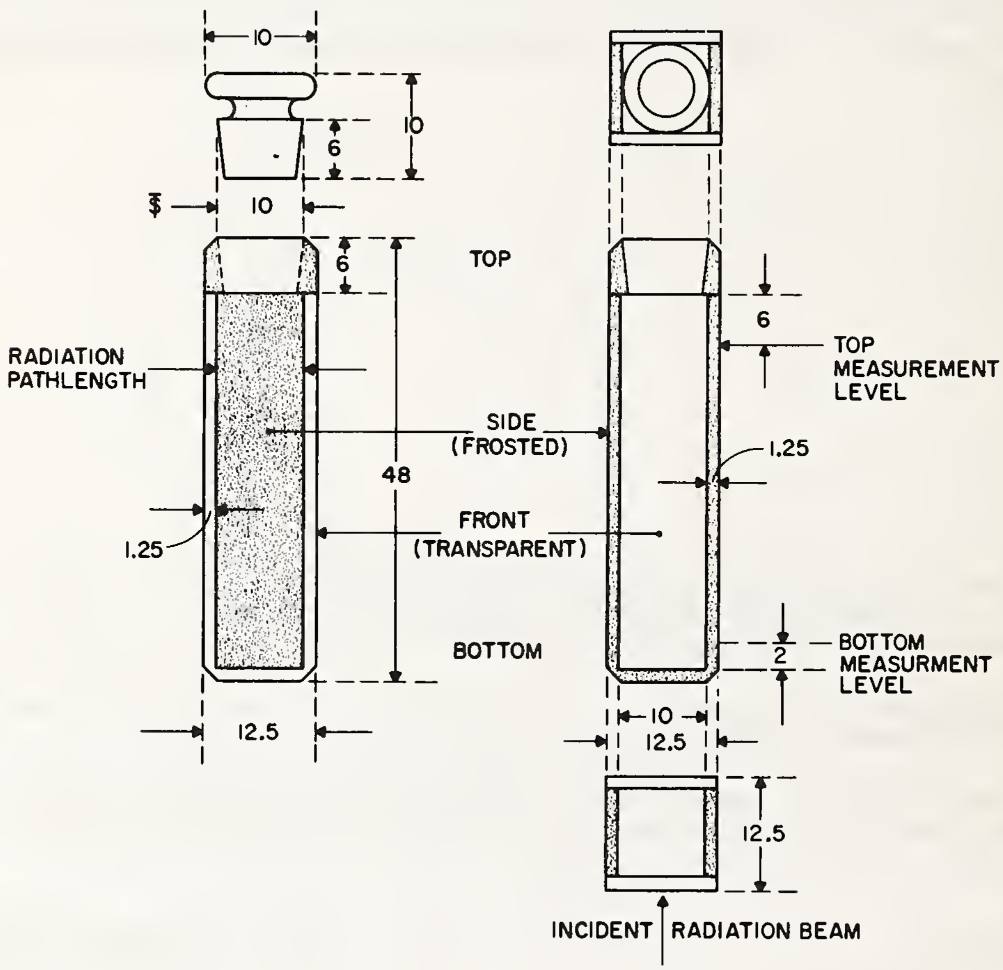
J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

The original cuvettes were designed and produced at the National Bureau of Standards using special techniques and non-fluorescent optical-quality fused silica as described in NBS SP 260-32 [1]. The transparent windows are attached to the body of the cuvette by direct fusion, and the unit was stress-relieved by annealing. The overall flatness of each surface of a transparent window is within two fringes (546 nm Hg line). The radiation pathlength measurements of the cuvette were performed using electronic feeler-gauge type instruments capable of a resolution of 5 parts in 10^6. The development and production of SRM 932 is a result of the combined efforts of the Center for Analytical Chemistry, the Center for Mechanical Engineering and Process Technology, and the Instrument Shops Division. E. P. Muth and E. I. Klein designed and assembled the cuvettes, respectively. Since January 1977, the cuvettes issued as SRM 932 have been prepared by Starna, Ltd., Dayenham, Essex, U. K., in accordance with NBS techniques and specifications described in NBS SP 260-32. The radiation pathlength measurements of all of these cuvettes, regardless of manufacturer, have been performed by E. G. Erber of the Mechanical Processes Division.

Reference:

[1] Mavrodineanu, R., and Lazar, J. W., Standard Reference Materials: Standard Quartz Cuvettes for High Accuracy Spectrophotometry, Nat. Bur. Stand. (U.S.), Spec. Publ. 260-32 (December 1973). Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, 55 cents.



U. S. Department of Commerce
Frederick B. Dent
Secretary

National Bureau of Standards
Richard W. Roberts, Director

National Bureau of Standards Certificate

Standard Reference Material 933

Clinical Laboratory Thermometer

This Standard Reference Material is intended for use in clinical laboratories as a primary calibrant, particularly in the area of clinical enzymology. It is comprised of three individually-calibrated thermometers. The certified corrections for each thermometer, as a computer print-out sheet, are attached, together with information relating to the calibrating details and to the International Practical Temperature Scale of 1968.

Each thermometer is a solid-stem, mercury-in-glass instrument 180 ± 5 mm in length, and marked with a 95 mm immersion line. The stems are plain front, enameled back made of lead-glass thermometer tubing 7 mm in diameter. Nitrogen gas fills the space above the mercury. Each thermometer has an auxiliary scale from –0.20 to + 0.20 °C with 0.05 °C divisions.The main scales of the thermometers are 24.00 to 26.00 °C, 29.00 to 31.00 °C, and 36.00 to 38.00 °C, respectively, with 0.05 °C divisions. Calibrated points are 0 °C, and 25, 30, or 37 °C depending on the scale of the individual thermometer. Detailed descriptions and drawings of these thermometers are given in the following publications:

Mangum, B. W., Clin. Chem. 20, 670 (1974).
Mangum, B. W., and Wise, J. A., NBS Special Publication 260-48, U. S. Government Printing Office, Washington, D.C. 20402, 1974.

The thermometers were manufactured by Princo Instruments, Inc. of Southampton. Pa. Measurements leading to the certification were performed by J. A. Wise of the Heat Division, Institute for Basic Standards.

The general direction of the technical work was performed by B. W. Mangum of the Heat Division, Institute for Basic Standards.

Washington, D.C. 20234
October 1, 1974

J. Paul Cali, Chief
Office of Standard Reference Materials

U. S. Department of Commerce
Frederick B. Dent
Secretary
National Bureau of Standards
Richard W. Roberts, Director

National Bureau of Standards
Certificate
Standard Reference Material 934
Clinical Laboratory Thermometer

This Standard Reference Material is intended for use in clinical laboratories as a primary calibrant, particularly in the area of clinical enzymology. The thermometer is individually calibrated at four points. The certified corrections, as a computer print-out sheet, are attached, together with information relating to the calibration details and to the International Practical Temperature Scale of 1968.

The thermometer is a solid-stem, mercury-in-glass instrument 300 ± 5 mm in length, and marked with a 95-mm immersion line. The stem is plain front, enameled back, made of lead-glass thermometric tubing 7 mm in diameter. Nitrogen gas fills the space above the mercury. It has an auxiliary scale from −0.20 to +0.20 °C with 0.05 °C divisions. The main scale extends from 24.00 °C to 38.00 °C in 0.05 °C divisions. The calibrated points are 0, 25, 30, and 37 °C. Detailed description and drawings of the thermometer are given in the following publications:

Mangum, B. W., Clin. Chem. 20, 670 (1974).
Mangum, B. W., and Wise, J. A., NBS Special Publication 260-48, U. S. Government Printing Office, Washington, D. C. 20402, 1974.

The thermometers were manufactured by Princo Instruments Inc., of Southampton, Pa.

Measurements leading to the certification were performed by J. A. Wise of the Heat Division, Institute for Basic Standards.

The general direction of the technical work was performed by B. W. Mangum of the Heat Division, Institute for Basic Standards.

Washington, D.C. 20234
October 23, 1974

J. Paul Cali, Chief
Office of Standard Reference Materials

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Acting Director

National Bureau of Standards
Certificate

Standard Reference Material 935
Crystalline Potassium Dichromate
for Use as an Ultraviolet Absorbance Standard

R. W. Burke and R. Mavrodineanu

This Standard Reference Material consists of crystalline potassium dichromate of established purity. Solutions of known concentrations of this SRM in 0.001 N perchloric acid are certified for their apparent* specific absorbances**, ϵ_a, at 23.5 °C.

This SRM is intended to be used as a reference standard for the verification of the accuracy and linearity of the absorbance scale of absorption spectrometers that can provide an effective spectral bandpass of 1.2 nm or less at 235 nm, and 0.8 nm or less at 257, 313, 345, and 350 nm. Such verification is accomplished by comparing the measured apparent absorbances, A_a, to the A_a calculated from the certified ϵ_a values as described under "Instructions for Use."

Table 1 gives the certified values of ϵ_a in $kg \cdot g^{-1} \cdot cm^{-1}$ for five concentrations of the SRM 935 potassium dichromate in 0.001 N perchloric acid at 23.5 °C and the indicated wavelengths and spectral bandpasses for a 1-cm internal pathlength.

Table 1. ϵ_a, Apparent Specific Absorbance,[a] $kg \cdot g^{-1} \cdot cm^{-1}$

Nominal Concentration $g \cdot kg^{-1}$	Wavelength and (Bandpass) nm 235.0(1.2)	257.0(0.8)	313.0(0.8)	345.0(0.8)[b]	350.0(0.8)	Uncertainty[c]
0.020	12.260	14.262	4.805	10.604	10.672	± 0.034
.040	12.304	14.318	4.811	10.603	10.682	± .020[d]
.060	12.347	14.374	4.816	10.602	10.692	± .020[d]
.080	12.390	14.430	4.821	10.601	10.701	± .020[d]
.100	12.434	14.486	4.827	10.600	10.711	± .020[d]

[a] ϵ_a values are given to the third decimal place to preserve the smooth variation of the data with concentration, although the uncertainties are in the second decimal place.

[b] Wavelength 345.0 nm is near one of the two isosbestic points in $HCrO_4^-/Cr_2O_7^=$ spectra. Because it is on the slope of the composite spectrum, reproduction of the ϵ_a values is dependent on wavelength accuracy. Measurements at this wavelength should be made only for verification of the linearity of the absorbance scale.

[c] ϵ_a values are not corrected for the effects of internal multiple reflections within the cuvette, nor have the weights been corrected to vacuum. With these two exceptions, the uncertainties given include all known systematic errors and the 95 percent confidence interval of the mean.

[d] At wavelength 313.0 nm, the uncertainty is reduced to ±0.010.

*The term "apparent" is used because no corrections have been applied to the data for the effects of internal multiple reflections within the cuvette or for buoyancy, i.e., the weights used to express concentrations have not been corrected to vacuum. These combined corrections do not exceed 0.2 percent. The specific absorbances are given in reference 1.

The nomenclature used in this certificate is that recommended by K. D. Mielenz, Anal. Chem. **48, 1093-1094 (1976), which is reproduced in the Appendix of NBS Special Publication 260-54.

Washington, D.C. 20234
June 1, 1977

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

The overall direction and coordination of the technical measurements leading to this certificate were performed under the joint chairmanship of I. L. Barnes and J. R. DeVoe.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. W. Seward.

We wish to acknowledge: The contributions of K. D. Mielenz, NBS Analytical Chemistry Division, for his many valuable discussions and suggestions, and H. H. Ku, NBS Applied Mathematics Division, for his statistical treatment of the data; the cooperation and early support of the National Institute of General Medical Sciences in the research leading to this SRM; and the encouragement of George N. Bowers, Jr., M.D., Hartford Hospital, Hartford, Conn., and Royden N. Rand, Ph.D., Eastman Kodak Co., Research Laboratories, Rochester, N.Y.

PREPARATION AND CERTIFICATION

The details of the preparation and certification of SRM 935 are provided in NBS Special Publication 260-54, Certification and Use of Acidic Potassium Dichromate Solutions as an Ultraviolet Absorbance Standard [2]. This publication should be referred to every time SRM 935 is to be used. Briefly, the transmittances, T, of the solutions prepared from the undried, as received, material were measured with the NBS Institute for Materials Research high-accuracy transmission spectrometer [3].

The ϵ_a values were calculated for each wavelength using the relation:

$$\epsilon_a = \frac{D_s - D_b}{b \times c} = \frac{A_a}{b \times c} \qquad (1)$$

where:

ϵ_a = apparent specific absorbance
A_a = apparent absorbance
D_s = transmittance density of the sample solution, $-\log_{10} T_s$
D_b = transmittance density of the blank solution, $-\log_{10} T_b$
b = internal cuvette pathlength, cm
c = concentration, by weight, of $K_2Cr_2O_7$ solution, $g \cdot kg^{-1}$

The crystalline potassium dichromate used for SRM 935 is a special lot of analytical reagent grade material obtained from the J. T. Baker Chemical Co., Phillipsburg, N.J.

Assay: A coulometric assay of the purity of the undried material was performed by G. Marinenko of the NBS Analytical Chemistry Division. The purity of SRM 935, expressed as an oxidant, was found to be 99.972 ± 0.005 percent where the uncertainty figure represents the 95 percent confidence interval for the mean based on 11 degrees of freedom. In addition, the material was examind by optical emission spectrometry for trace elemental impurities by J. A. Norris of the NBS Analytical Chemistry Division. The only significant impurities detected were sodium and rubidium. Their concentrations were estimated to be in the range of 0.02 and 0.03 percent, respectively. Drying at 105 °C for 12 hours showed that the surface moisture of this material was less than 0.01 percent.

Stability: Solutions prepared from SRM 935 in the concentration range indicated in table 1 and made according to the instructions given in NBS SP 260-54 have been found to be stable within the uncertainties given in table 1 for at least six months when stored at room temperature and protected from evaporation and exposure to light.

INSTRUCTIONS FOR USE

The use of SRM 935 as an absorbance standard requires the careful preparation of a series of solutions of known concentrations, c, of the potassium dichromate in 0.001 N perchloric acid. These solutions are transferred to a quartz cuvette of known pathlength, b, and their apparent absorbances measured at wavelengths 235, 257, 313, and 350 nm, using the spectral bandpass requirements given in table 1. The preparation and measurement of these solutions are described in detail in Section 5 of NBS SP 260-54.

The accuracy of the absorbance scale of the spectrometer being tested is ascertained by comparing the measured apparent absorbances, A_a, of a series of 0.001 N perchloric acid solutions containing 0.020 to 0.100 gram $K_2Cr_2O_7$/kg to the A_a values calculated from the certified ϵ_a values. Although the ϵ_a data in table 1 are given for nominal concentrations of 0.020, 0.040, 0.060, 0.080, and 0.100 g $K_2Cr_2O_7$/kg, the ϵ_a values for concentrations between these nominal concentrations can be determined by linear interpolation. Using the appropriate ϵ_a values, the calculated A_a values at 23.5 °C are obtained from the expression:

$$A_a = \epsilon_a \times b \times c \qquad (2)$$

Calculations:

An example of the calculation of A_a for one concentration of $K_2Cr_2O_7$ under a specified set of conditions is shown below. Calculations of A_a for other concentrations and wavelengths are performed in a similar manner.

Conditions: Wavelength = 350 nm, spectral bandpass 0.8 nm or less
$b = 0.9982$ cm
$c = 0.04375\ g \cdot kg^{-1}$
$t = 23.5$ °C

From column 6, table 1, the ϵ_a for concentrations of 0.040 and 0.060 $g \cdot kg^{-1}$ are 10.682 and 10.692, respectively. The corresponding ϵ_a for $c = 0.04375\ g \cdot kg^{-1}$ is:

$$\epsilon_a = 10.682 + \frac{0.04375 - 0.040}{0.060 - 0.040}(10.692 - 10.682)$$

$$\epsilon_a = 10.682 + 0.0019$$

$$\epsilon_a = 10.684$$

The calculated apparent absorbance, A_a, from equation 2, is:

$$A_a = 10.684 \times 0.9982 \times 0.04375$$

$$A_a = 0.4666$$

The uncertainty, ΔA_a, in the calculated A_a is determined from the combined uncertainties in ϵ_a, b, and c in equation 2, provided no other systematic errors are present. Thus:

$$\Delta A_a = bc\left|\Delta\epsilon_a\right| + \epsilon_a c\left|\Delta b\right| + \epsilon_a b\left|\Delta c\right| \qquad (3)$$

To evaluate ΔA_a, $\Delta\epsilon_a$ is taken from column 7 of table 1 and the Δb and Δc values must be determined experimentally.

In the experiments performed to obtain the ϵ_a values in table 1, the uncertainties for b and c did not exceed 1 part in 10^4 and 2 parts in 10^4, respectively.

The solution of equation 3 gives:

$$\Delta A_a = 1(0.044)\ (0.020) + 10.7(0.044)\ (0.0001) + 10.7(1)\ (0.0000088)$$
$$= 0.0010$$

Thus, the uncertainty of A_a, for the above set of conditions, is ±0.0010.

The correction of the absorbance scale of the absorption spectrometer under test is determined by plotting the differences between A_a measured and A_a calculated as a function of absorbance. A typical plot of such a graph is shown in figure 1. The apparent absorbances measured on this instrument at 350 nm are accurate when the indicated correction is subtracted from the corresponding absorbance scale reading, provided that the conditions of wavelength accuracy, spectral bandpass, and absence of stray light are fulfilled as specified in NBS SP 260-54. Correction curves for wavelengths 235, 257, and 313 nm are obtained in a similar manner.

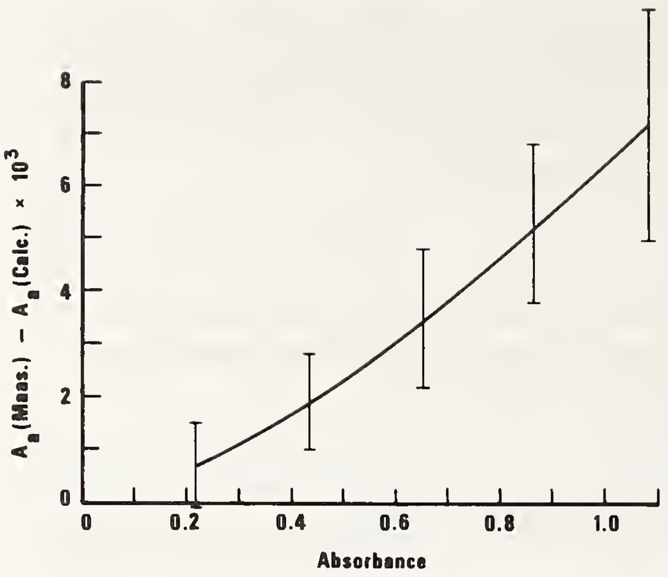


Figure 1. Correction curve for the absorbance scale of a precision spectrometer. The error bars are the sum of the errors arising from the uncertainties in the certified apparent specific absorbances, ϵ_a, cuvette pathlength, b, and concentration, c.

Temperature Correction:

Although ϵ_a values in table 1 are certified at 23.5 °C, SRM 935 can be used as an absorbance standard at other temperatures in the range 20 to 30 °C provided corrections are made to the ϵ_a values. Over this range the apparent specific absorbances decrease linearly with increasing temperature for all the wavelengths given in table 1. The corresponding temperature coefficients, k, for these wavelengths are given in table 2.

Table 2. Variation of ϵ_a with Temperature Over the Range 20 to 30 °C.

λ, nm	Temperature Coefficient, k Percent per degree Celsius
235	−0.05
257	−0.05
313	−0.02
345	−0.08
350	−0.05

The value of ϵ_a at any temperature in the range 20 to 30 °C can be calculated from the certified value and the appropriate temperature coefficient using the relation:

$$\epsilon_a^t = \epsilon_a^{23.5}\left[1 + \frac{k}{100}(t - 23.5)\right]$$

where: ϵ_a^t = apparent specific absorbance at temperature t (°C)

$\epsilon_a^{23.5}$ = apparent specific absorbance certified at 23.5 °C.

k = temperature coefficient, percent per °C.

REFERENCES

1. Burke, R. W., and Mavrodineanu, R., Acidic Potassium Dichromate Solutions as Ultraviolet Absorbance Standards, J. Res. Nat. Bur. Stand. (U.S.), **80A** (Phys. and Chem.), No. 4, 631-636 (July-Aug. 1976).
2. Burke, R. W., and Mavrodineanu, R., Certification and Use of Acidic Potassium Dichromate Solutions as an Ultraviolet Absorbance Standard, NBS Spec. Publ. 260-54 (1977). Copies may be obtained from the Office of Standard Reference Materials, National Bureau of Standards, Washington, D.C. 20234.
3. Mavrodineanu, R., An Accurate Spectrophotometer for Measuring the Transmittance of Solid and Liquid Materials, J. Res. Nat. Bur. Stand. (U.S.), **76A** (Phys. and Chem.), No. 5, 405-425 (1972).

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 936

Quinine Sulfate Dihydrate

R. A. Velapoldi and K. D. Mielenz

This Standard Reference Material is intended for use in the evaluation of methods and the calibration of fluorescence spectrometers. It is certified for the relative molecular emission spectrum, E(λ), in radiometric units for a solution of 1.28 x 10^{-6} mol/L quinine sulfate dihydrate in 0.105 mol/L perchloric acid using an excitation wavelength of 347.5 nm. The certified values of the molecular emission spectrum at 5 nm wavelength intervals from 375 to 675 nm are given in table 1. These values have been corrected for instrument and sample parameters, including the spectral responsivity of the detection system, monochromator bandwidth, photomultiplier tube nonlinearity, monochromator wavelength error, solvent refractive index, and cell window transmittance. The relative standard error in E(λ), RSE [E(λ)], is given in table 1. The estimate of the relative systematic error limits in the molecular emission spectrum, RSEL [E(λ)], is also given in table 1 and was determined by the addition of the absolute values of the estimated systematic errors. These relative error limits include uncertainties in the calibration values for the spectral responsivity, the wavelength position of the emission peak maximum, and in the corrections applied for instrument and sample parameters.

From the certified values of E(λ), values may be calculated for the molecular emission spectrum in the various photon, radiometric, wavelength, and wavenumber units using the following equation: [1,2]

$$E(\lambda) = \frac{E_p(\lambda)}{\lambda} = \frac{E(\tilde{\nu})}{\lambda^2} = \frac{E_p(\tilde{\nu})}{\lambda^3}$$

These values have been calculated and are given in NBS Special Publication 260-64.

The technical emission spectrum, $E^T(\lambda)$, i.e., the emission spectrum corrected for instrument parameters only, is also given in SP 260-64. The quinine sulfate dihydrate used for SRM 936 was a special lot of material obtained from the J. T. Baker Chemical Co., Phillipsburg, N.J.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears and R. W. Seward.

Washington, D.C. 20234
April 1, 1979

George A. Uriano, Chief
Office of Standard Reference Materials

(over)

Table 1. The Molecular Emission Spectrum, E(λ), of Quinine Sulfate Dihydrate in 0.105 mol/L $HClO_4$, the Relative Standard Error, RSE, and the Estimated Relative Systematic Error Limits, RSEL, in the E(λ) Values.

λ,nm	E(λ)	RSE [E(λ)]	RSEL [E(λ)]	λ,nm	E(λ)	RSE [E(λ)]	RSEL [E(λ)]
375.0	0.005	0.019	0.087	525.0	0.302	0.001	0.029
380.0	.012	.006	.078	530.0	.264	.003	.029
385.0	.028	.003	.071	535.0	.231	.003	.029
390.0	.057	.003	.064	540.0	.201	.002	.029
395.0	.103	.002	.059	545.0	.175	.002	.029
400.0	.170	.002	.054	550.0	.153	.001	.029
405.0	.257	.003	.049	555.0	.132	.001	.029
410.0	.359	.003	.045	560.0	.116	.001	.029
415.0	.471	.003	.041	565.0	.101	.002	.029
420.0	.586	.003	.037	570.0	.088	.002	.029
425.0	.694	.003	.034	575.0	.076	.003	.029
430.0	.792	.002	.031	580.0	.065	.003	.029
435.0	.874	.002	.028	585.0	.057	.001	.029
440.0	.940	.001	.026	590.0	.050	.003	.030
445.0	.984	.001	.024	595.0	.043	.004	.030
450.0	.999	.001	.023	600.0	.037	.006	.030
455.0	.997	.001	.023	605.0	.032	.002	.030
460.0	.982	.001	.024	610.0	.028	.006	.030
465.0	.947	.001	.024	615.0	.024	.003	.030
470.0	.897	.001	.025	620.0	.021	.011	.030
475.0	.838	.002	.026	625.0	.018	.003	.030
480.0	.782	.002	.027	630.0	.016	.015	.030
485.0	.719	.002	.027	635.0	.014	.014	.030
490.0	.657	.002	.027	640.0	.011	.037	.030
495.0	.595	.003	.027	645.0	.010	.015	.030
500.0	.541	.002	.027	650.0	.009	.027	.030
505.0	.486	.001	.028	655.0	.008	.035	.031
510.0	.434	.003	.028	660.0	.007	.073	.031
515.0	.386	.003	.028	665.0	.006	.046	.032
520.0	.342	.002	.028	670.0	.005	.053	.032
				675.0	.004	.065	.033

SUPPLEMENTARY DATA

The following data for the specific molar absorbances, water content, photon yields, and fluorescence lifetimes are considered to be supplementary and are not to be considered certified values.

The quinine sulfate dihydrate (QSD) used for SRM 936 was found to be homogeneous to better than 0.5% by thin-layer chromatography with development by two solvent systems and the determination of specific molar absorbances, ϵ, at three different wavelengths. The SRM contains approximately 1.7% of an impurity as determined by high performance liquid chromatography using absorbance and fluorescence detection. This impurity is believed to be dihydroquinine sulfate dihydrate, which has optical characteristics that are similar to those of the quinine sulfate dihydrate. The ultraviolet absorption spectrum of SRM 936 in 0.105 mol/L $HClO_4$ exhibits the following absorption maxima:

$$250.0 \text{ nm}, \epsilon_{max} = 56{,}990 \pm 90 \text{ L·mol}^{-1}\text{·cm}^{-1}$$

$$347.5 \text{ nm}, \epsilon_{max} = 10{,}810 \pm 20 \text{ L·mol}^{-1}\text{·cm}^{-1}$$

and, on the side of a peak:

$$365.0 \text{ nm}, \epsilon_{obs} = 6{,}920 \pm 10 \text{ L·mol}^{-1}\text{·cm}^{-1}$$

The water content of this material was measured by two methods. The average of six determinations by the Karl-Fischer method gave a value of (4.74 ± 0.05%), while the average of four determinations by a weight loss procedure gave a value of (4.57 ± 0.04%). The theoretical value for water in quinine sulfate dihydrate is 4.60%.

The photon yield, Q, and the fluorescence lifetime, τ, of SRM 936 were compared to values obtained for a sample of purified quinine sulfate dihydrate and are summarized below:

	Q 0.5 mol/L H_2SO_4	τ, ns 0.5 mol/L H_2SO_4
SRM 936, QSD	0.544 ± 0.03	19.1 ± 0.1
Purified QSD	0.546[a]	19.2 ± 0.1

[a]Melhuish, W. H., J. Phys. Chem. **65**, 229 (1961); *ibid,* New Zealand J. Sci. Tech. **37**, 142 (1955).

PREPARATION AND USE OF SRM 936

This Standard Reference Material is for "in vitro" diagnostic use as a clinical laboratory standard. A "stock" standard solution containing 0.1 mg/mL of quinine sulfate may be prepared as follows: Weigh 0.100 g of SRM 936 to the nearest one-tenth milligram and quantitatively transfer it to a 1000-mL volumetric flask. Dilute to the calibrated volume with 0.105 mol/L $HClO_4$, to give a solution that is 1.28×10^{-4} mol/L (0.1 mg/mL) in quinine sulfate. Store this solution in the dark in a well-stoppered, glass bottle. A "working" standard solution containing 1 μg/mL may be prepared by transferring 10 mL of the above "stock" standard solution to a 1000-mL volumetric flask and diluting to the calibrated volume with 0.105 mol/L $HClO_4$ to give a solution that is 1.28×10^{-6} mol/L (1 μg/mL) in quinine sulfate. Store this solution in the same manner as the above "stock" standard solution.

Several opinions regarding the stability of quinine sulfate solutions have appeared in the literature [3]. NBS considers the 0.1 mg/mL "stock" standard solution prepared from SRM 936 to be stable for 3 months when stored as specified; and the 1 μg/mL "working" standard solution to be stable for 1 month when so stored.

SRM 936 should be kept in its original bottle and stored in the dark at room temperature (30 °C or less). It should not be subjected to heat or direct sunlight during storage. Experience at NBS indicates that under proper storage this material is stable for at least 3 years. If this material degrades beyond the limits certified, purchasers will be notified by NBS. It is recommended that the material not be used after 3 years from the date of purchase.

References:

[1] Ejder, E. J., J. Opt. Soc. Amer. **59,** 223 (1969).

[2] Melhuish, W. H., J. Res. Nat. Bur. Stand. (U.S.) **76A,** No. 6, 547 (1972).

[3] Melhuish, W. H., J. Phys. Chem. **65,** 229 (1961); Gill, J. E., Photochem. and Photobiol. **9,** 313 (1969); Birks, J. B., J. Res. Nat. Bur. Stand. (U.S.) **80A,** 389 (1976); Heller, C. A., Henry, R. A., McLaughlin, B. A., and Bless, D. E., J. Chem. Eng. Data **19,** 214 (1974); West, M. A., and Kemp, D. R., Int'l. Lab., p. 27 (May/June 1976); and White, J. U., Pittsburgh Conf. Abstracts, Paper 488 (1977).

This Standard Reference Material has been measured and certified at the laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D.C. 20234

The date of issuance and certification of SRM 936 was April 1, 1979

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 937

Iron Metal

(Clinical Standard)

This Standard Reference Material is certified as a material of known purity for use as an assay standard for iron. It is intended primarily for the calibration of instrumentation and standardization of procedures employed in clinical analysis and for the routine evaluation of daily working standards used in these procedures.

Assay 99.90 ± 0.02 percent by weight

The assay shown is based on dissolution of representative samples in hydrochloric acid followed by reduction of the iron with stannous chloride and oxidation of the excess stannous chloride with mercuric chloride. Most of the iron was oxidized with a known weight of potassium dichromate and the remaining iron was titrated with potassium dichromate solution. The estimated uncertainty shown is based on judgment and includes allowances for known sources of possible error.

SRM 937 is electrolytic grade iron. Chemical analyses made on representative samples showed the presence of the following elements at the indicated concentrations in percent by weight: nickel, 0.041%; silicon, 0.008%; carbon, chromium, and cobalt, each 0.007%; copper, manganese, oxygen, and sulfur, each 0.006%; molybdenum, 0.005%; phosphorous, 0.003%; germanium and nitrogen, each 0.001%; with the total of all other elements <0.003%.

The iron content, by difference, is 99.89+%, which is in agreement with the assay value.

The chemical assay was performed by B. I. Diamondstone; the elemental determinations by R. Alvarez, J. R. Baldwin, E. Belkas, B. S. Carpenter, M. M. Darr, E. R. Deardorff, T. E. Gills, L. A. Machlan, E. J. Maienthal, L. J. Moore, C. W. Mueller, T. J. Murphy, P. J. Paulsen, K. M.Sappenfield, B. A. Thompson, and S. A. Wicks.

The overall direction and coordination of the technical measurements leading to certification were under the chairmanship of I. L. Barnes.

The technical and support aspects concerning preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, D.C. 20234
June 9, 1978

J. P. Cali, Chief
Office of Standard Reference Materials

(over)

This Standard Reference Material is intended for "in vitro" diagnostic use only.

This material is intended for use as a standard for iron determination in clinical chemistry.

Preparation of stock iron standard solution (0.02 mmol/mL)

Weigh approximately 1 g of SRM 937 to the nearest 0.1 mg and transfer it to a one-liter volumetric flask. Dissolve the metal in 100 mL of 6 mol/L HCl and dilute to one liter with water

The exact concentration of this stock solution, in mmol/mL, is expressed by:

$$\frac{\text{mg (SRM 937)} \times 0.999}{55.847 \times 1000}$$

Preparation of working stock iron standard solution (0.2 μmol/mL)

Transfer 10 mL of the stock iron standard solution to a one-liter volumetric flask and add 0.2 mol/L HCl to the graduation line.

Preparation of working iron standard solutions

Prepare more dilute solutions by pipetting known volumes of the working stock iron standard solution into volumetric flasks and diluting to the graduation line with 0.2 mol/L HCl. These solutions should be prepared daily. [Note: In the preparation of very dilute solutions, the 0.2 mol/L HCl may contain sufficient iron, as an impurity, to affect the calculated iron concentrations of these dilute solutions.]

Precautions: All volumetric glassware used should conform to the specifications for Class A glassware and, for highest accuracy, should be individually calibrated and used at the calibration temperature [1]. All glassware should be cleaned in hot dilute HCl and rinsed in distilled water.

This SRM should be stored in the tightly closed original bottle under normal laboratory conditions.

Reference:

[1] J. Lembeck, The calibration of small volumetric laboratory glassware, NBSIR 74-461, National Bureau of Standards, Washington, D.C. 20234.

The date of issuance and certification of this Standard Reference Material was June 9, 1978.

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Acting Director

National Bureau of Standards
Certificate
Standard Reference Material 1968
Gallium
Melting-Point Standard

Cell No. ___________

29.7723 ± 0.0007 °C
International Practical Temperature Scale of 1968

This Standard Reference Material is intended to be used to calibrate thermometers near 30 °C. SRM 1968 consists of approximately 25 grams of high-purity gallium in a specially designed, epoxy-sealed cell. The melting-point temperature of this cell,as given above, was verified using a set of three stable thermistors that were regularly recalibrated against a Standard Platinum Resistance Thermometer. The Standard Platinum Resistance Thermometer, which was recently calibrated on IPTS-68 at NBS, was used in the determination of the gallium triple-point temperature at NBS. The stated uncertainty, ±0.0007 °C, represents one-half of the total range of the temperatures observed in 152 determinations of the melting points of this and similar cells.

SRM 1968 can be used to calibrate all thermometers that have temperature sensing elements smaller than 3.5 mm in diameter. With care, such thermometers may be calibrated with an accuracy of ±0.0007 °C. The liquid-solid equilibrium temperature of pure gallium at a pressure of one standard atmosphere (101.3 kPa) is 29.7723 ± 0.0004 °C [1]. This equilibrium is best realized by slowly melting the solid.

The high-purity gallium metal used in SRM 1968 was obtained from Eagle-Picher Industries, Inc., Quapaw, Oklahoma.

The technical measurements at NBS leading to certification were performed by D. D. Thornton and B. W. Mangum of the NBS Heat Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. W. Seward.

Washington, D.C. 20234
June 1977

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

SRM 1968 was designed by D. D. Thornton of the NBS Heat Division. The cell components were fabricated by the NBS Instrument Shops Division. The cell consists of a Teflon body, a nylon well, and a nylon cap stem, as shown in figure 1. Filling, assembling, and sealing of the cells were performed in a dry argon atmosphere.

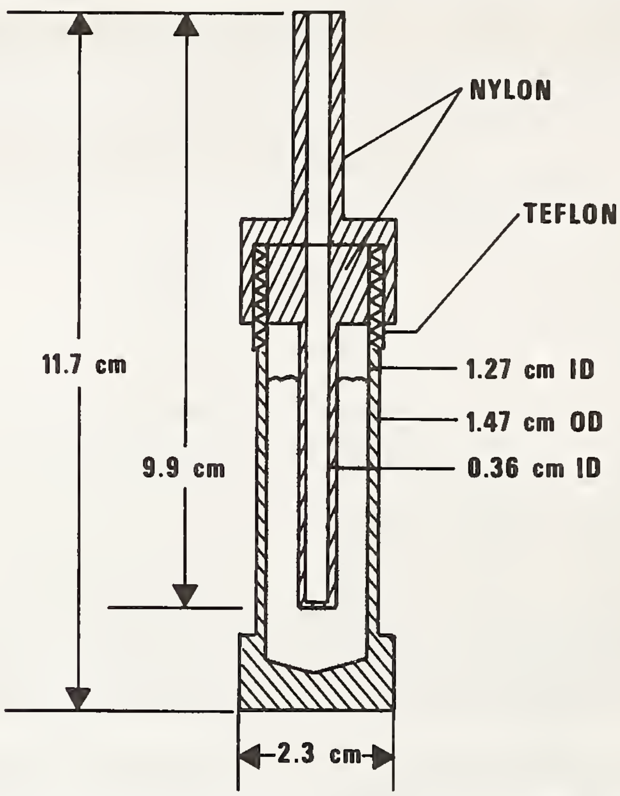


Fig. 1. Cross-sectional drawing of SRM 1968, Gallium Melting-Point Standard.

CAUTION: Because the nylon parts of the cell will absorb water, the cell should not be immersed in water for periods exceeding two days. Particular care should be exercised to prevent water entering the cap stem and well. When the cell is immersed in water, at least one centimeter of the cap stem should protrude from the surface.

NOTE: The gallium in SRM 1968 was frozen before leaving NBS. If the metal should melt completely, it should refreeze readily in an ambient of 20 °C. Should the metal supercool and remain liquid at room temperature, solidification can be induced by placing the cell in an ice bath or freezer for about two hours.

Instructions for Use of SRM 1968

SRM 1968 should be used as a calibrant in a temperature-regulated bath. To calibrate a thermometer with SRM 1968, the user should employ a light mineral oil for thermal contact in the well. Any water should be removed from the sensor before inserting it into the well. The temperature sensing element should be inserted into the well and the cell then inserted into the bath, which has been regulated to a temperature of 30.0 ± 0.1 °C. Within 30 to 60 minutes, the thermometer should indicate a steady reading, which corresponds to the gallium melting point (29.7723 °C).The duration of the melting plateau depends on the bath temperature (figure 2) Thus, once melting has begun, the duration of the plateau can be prolonged by setting the bath to 29.80 ± 0.03 °C–just above the gallium melting point. The bath temperature can be checked by temporarily placing the thermometer sensor directly into the bath.

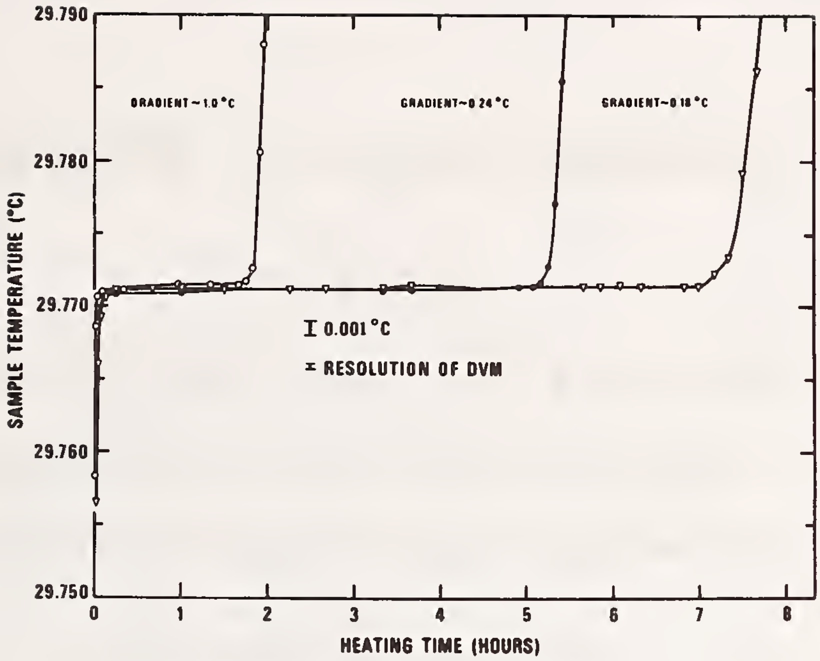


Fig. 2. Melting curves of SRM 1968 (nominal 99.99999% pure gallium) for several different temperature gradients.

Accuracy of Calibration

The melting temperature of the gallium is very accurately known; however, several factors introduce uncertainty, and thus reduce the accuracy of the calibration. The most likely limiting factor in the accuracy of the calibration is the scale (or read-out) of the thermometer being calibrated. For example, a thermometer with a digital read-out to the nearest 0.01 °C will read all temperatures from 29.765 to 29.774 °C as 29.77 °C. For thermometers with resistive sensing elements (e.g., thermistors), another limiting factor is the self-heating caused by the current through the element. The size of the self-heating effect depends upon the local environment of the sensing element, and will introduce an uncertainty into the calibration. The instruction manual for the thermometer should be consulted, both in this regard and in reference to the resolution problem mentioned above.

The immersion effect is yet another potential source of calibration error. In the presence of a temperature difference (ΔT) between the bath and the sensor, heat will be conducted along the leads to or from the sensor and will produce an immersion error (δT). When the thermometer is sufficiently immersed, most of the heat conducted along the leads is transferred through the insulation and the immersion error, δT, is insignificant. Because the error, δT, depends on ΔT, any significant immersion error can be minimized. The extent of such a problem can be determined when calibrating a thermometer with SRM 1968 in a bath at 30.0 °C. Set the bath temperature to 31.0 °C and observe the thermometer in the gallium cell for 15 minutes. When a new steady value is reached, the difference (δT) between this value and the value obtained with the bath at 30.0 °C is a measure of the immersion effect. For example, if the thermometer showed a change of 0.02 °C when the bath temperature was increased by 1 °C, then the immersion effect is 0.02 °C per degree Celsius. This example implies that the bath must be set no higher than about 29.9 °C if the immersion error is to be less than 0.003 °C.

[1] Mangum, B. W., and Thornton, D. D., Determination of the Triple-Point Temperature of Gallium (to be published).

[2] Thornton, D. D., The Gallium Melting-Point Standards: A Determination of the Liquid-Solid Equilibrium Temperature of Pure Gallium on the International Practical Temperature Scale of 1968, Clinical Chemistry **23**, No. 4, 719-724 (April 1977).

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 2009

Didymium Glass Filter for Checking the Wavelength Scale of Spectrophotometers

Kenneth L. Eckerle and William H. Venable, Jr.

Serial Number:

This SRM is intended for use in calibrating the wavelength scale in the visible wavelength region of scanning spectrophotometers having nominal bandwidths in the range 1.5 to 10.5 nm. Depending upon the bandwidth of the spectrophotometer, anywhere from 14 to 24 wavelength corrections can be determined from 400 to 760 nm. Detailed instructions on the use of this SRM and examples of its use are given in NBS Special Publication 260-66. Each didymium-glass filter is identified by the SRM number and a serial number.

The wavelengths of the transmittance minima as obtained from measurements on two filters representative of the melt are given in Table 1. These values are given for seven equally spaced values of the half-height width of triangular passbands. The minima number is identified in the figure that illustrates the spectral transmittance as a function of wavelength. The wavelength values of nine points of inflection on the spectral transmittance curve as obtained on two filters are given in Table 2. These inflection points are representative of the melt and are also identified in the figure. These inflection points should only be used with the transmittance minima as described in Sections 2.2 and 2.3 in SP 260-66.

The measurements on which these tables are based were made at 25 °C with a high-precision reference spectrophotometer that has a wavelength accuracy of 0.04 nm. Table 3 indicates the estimated random (as obtained from 4 sets of measurements on a single filter) and systematic errors of the transmittance minima given in Table 1. Table 2 also indicates the range of the measured wavelengths of the inflection points. Trial calibrations made on several instruments, using both minima and inflection points, indicate that wavelength corrections made with these SRM's can be accurate to 0.2 nm. The uncertainty of a calibration, however, will depend upon the stability and other characteristics of a particular instrument.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Office of Standard Reference Materials by R. K. Kirby.

The spectral transmittance as a function of wavelength for a filter representative of the melt is given in Table 4. These values are not certified but are provided for use as specified in SP 260-66. *They should not be used to check the photometric scale of a spectrophotometer.*

It is recommended that the filter be handled only by its edges and when not in use it should be stored in the box provided. If cleaning is necessary, wet the filter with water and rub gently with optical lens tissue soaked with a mild soap solution, rinse with distilled water, rinse with isoprophyl alcohol, and rinse again with distilled water. Dry after each rinsing by wiping lightly with optical lens tissue.

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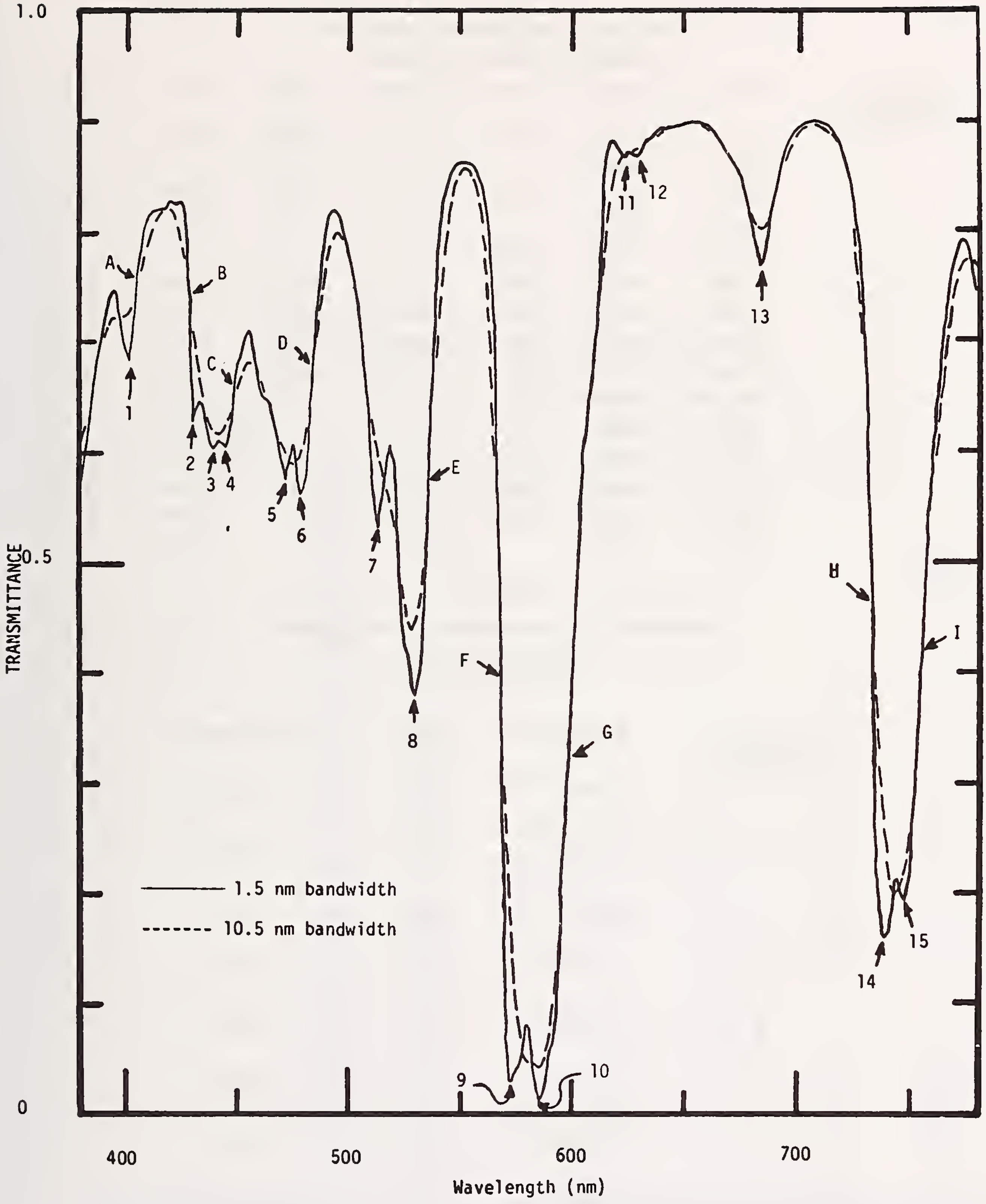
January 8, 1980

(over)

George A. Uriano, Chief
Office of Standard Reference Materials



Spectral transmittance of a typical didymium glass filter. Numbers indicate the principal points of minimum transmittance and letters indicate the principal points of inflection.

Table 1

Certified Wavelengths (nm) of the Transmittance Minima for the Indicated Bandwidths

Bandwidth Minimum No.	1.5 nm	3.0 nm	4.5 nm	6.0 nm	7.5 nm	9.0 nm	10.5 nm
1	402.42	401.81	401.69	401.66	401.42	400.95	
2	431.50	432.48					
3	440.27	440.52	441.84	442.52	442.37	442.08	441.33
4	445.59	445.14					
5	472.72	472.58	472.88				
6	478.89	479.34	479.28	478.31	477.36	476.50	475.65
7	513.45	513.61	513.89	514.31	515.38		
8	529.58	530.02	529.90	529.47	529.27	529.12	528.88
9	572.69	573.27	574.21	575.11	576.59		
10	585.34	585.54	585.77	586.02	585.99	585.35	584.42
11	623.62	624.02					
12	629.53	629.41	628.56	627.03	627.02		
13	684.66	684.68	684.71	684.72	684.71	684.66	684.58
14	739.86	739.96	740.24	740.91	742.01	742.97	743.65
15	748.28	748.10					

Table 2

Wavelengths and Transmittances at Nine Selected Points of Inflection

Point Identification	Wavelength (nm)	Range* (nm)	Transmittance†
A	406.44	+.08 −.06	0.7760
B	429.43	+.05 −.05	.7359
C	449.49	+.06 −.06	.6516
D	484.84	+.10 −.15	.6758
E	536.50	+.06 −.09	.5805
F	568.15	+.08 −.04	.4023
G	599.05	+.05 −.07	.3348
H	733.39	+.06 −.03	.4719
I	756.45	+.01 −.02	.4177

*The range of wavelengths within which the wavelength for the given transmittance will fall for symmetric triangular passbands with half-height bandwidths from 1.5 to 10.5 nm.

†These values of transmittance are not certified.

Table 3

Estimated Random and Systematic Errors of the Transmittance Minima

Band Number	Nominal Wavelength of Minimum Transmittance	Standard Deviation for Indicated Bandwidth						
		1.5 nm	3.0 nm	4.5 nm	6.0 nm	7.5 nm	9.0 nm	10.5 nm
1	402 nm	0.015 nm (0.07)†	0.018 nm	0.020 nm	0.021 nm	0.025 nm	0.029 nm	–
2	431	0.013 (0.06)	0.010	–	–	–	–	–
3	440	0.021 (0.05)	0.013	0.050	0.028	0.009	0.005	0.007 nm (0.25)
4	446	0.023 (0.06)	0.029	–	–	–	–	–
5	473	0.011 (0.08)	0.012	0.032	–	–	–	–
6	479	0.015 (0.15)	0.013	0.009	0.009	0.009	0.014	0.017 (0.25)
7	573	0.022 (0.08)	0.016	0.014	0.013	0.010	–	–
8	530	0.012 (0.21)	0.010	0.010	0.011	0.010	0.011	0.010 (0.25)
9	573	0.004 (0.05)	0.010	0.010	0.012	0.014	–	–
10	585	0.007 (0.06)	0.004	0.007	0.008	0.008	0.007	0.007 (0.25)
11	624	0.058 (0.06)	0.061	–	–	–	–	–
12	630	0.210 (0.02)	0.120	0.171	0.133	0.091	–	–
13	685	0.019 (0.05)	0.029	0.024	0.017	0.014	0.014	0.012 (0.25)
14	740	0.009 (0.25)	0.013	0.011	0.010	0.009	0.009	0.010 (0.25)
15	748	0.020 (0.04)	0.016	–	–	–	–	–

†Values in parentheses are estimates of the systematic error. The estimates for the 1.5 nm bandwidth were obtained from the data taken at 1.5 nm intervals as compared to data taken at 0.15 nm intervals. The method of estimating the systematic errors for the 10.5 nm bandwidth is described in Section 3.4 in SP 260-66.

TABLE 4

Transmittance (T) as a function of wavelength from 380 to 780.5 nm for a bandwidth of 1.5 nm. The estimates of random and systematic errors are also provided. These data are not certified.

Wave-Length (nm)	T	ΔT Standard Error	ΔT Systematic	Wave-Length (nm)	T	ΔT Standard Error	ΔT Systematic
380.00	.61832	.000054	.00010	381.50	.63896	.000085	.00010
383.00	.66210	.000064	.00010	384.50	.68291	.000079	.00010
386.00	.70112	.000047	.00010	387.50	.71885	.000071	.00010
389.00	.73590	.000095	.00010	390.50	.74954	.000040	.00010
392.00	.76201	.000083	.00010	393.50	.77104	.000055	.00010
395.00	.77620	.000064	.00010	396.50	.77123	.000038	.00010
398.00	.75013	.000069	.00010	399.50	.72419	.000079	.00010
401.00	.70966	.000070	.00010	402.50	.69505	.000071	.00010
404.00	.73683	.000057	.00010	405.50	.75886	.000047	.00010
407.00	.77756	.000088	.00010	408.50	.80673	.000072	.00010
410.00	.82277	.000058	.00010	411.50	.82813	.000038	.00010
413.00	.83115	.000060	.00010	414.50	.83287	.000075	.00010
416.00	.83222	.000060	.00010	417.50	.83200	.000029	.00010
419.00	.83277	.000059	.00010	420.50	.83844	.000066	.00010
422.00	.83711	.000064	.00010	423.50	.83328	.000052	.00010
425.00	.83511	.000049	.00010	426.50	.83414	.000045	.00010
428.00	.81620	.000055	.00010	429.50	.72877	.000023	.00010
431.00	.62212	.000096	.00010	432.50	.63478	.000039	.00010
434.00	.64183	.000057	.00010	435.50	.63392	.000059	.00010
437.00	.61858	.000049	.00010	438.50	.60090	.000038	.00010
440.00	.59163	.000064	.00010	441.50	.59496	.000053	.00010
443.00	.60082	.000063	.00010	444.50	.59576	.000068	.00010
446.00	.59315	.000055	.00010	447.50	.60769	.000044	.00010
449.00	.63551	.000083	.00010	450.50	.66313	.000060	.00010
452.00	.68002	.000069	.00010	453.50	.69110	.000057	.00010
455.00	.70206	.000027	.00010	456.50	.70341	.000033	.00010
458.00	.69051	.000025	.00010	459.50	.66807	.000049	.00010
461.00	.64890	.000057	.00010	462.50	.63966	.000060	.00010
464.00	.63837	.000070	.00010	465.50	.63353	.000040	.00010
467.00	.62094	.000072	.00010	468.50	.60572	.000053	.00010
470.00	.59042	.000008	.00010	471.50	.57213	.000072	.00010
473.00	.56160	.000033	.00010	474.50	.58414	.000031	.00010
476.00	.59528	.000052	.00010	477.50	.56003	.000052	.00010
479.00	.54953	.000070	.00010	480.50	.55659	.000036	.00010
482.00	.57326	.000025	.00010	483.50	.62009	.000040	.00010
485.00	.67764	.000052	.00010	486.50	.72126	.000072	.00010
488.00	.75412	.000045	.00010	489.50	.78321	.000082	.00010
491.00	.80556	.000035	.00010	492.50	.81759	.000060	.00010
494.00	.82117	.000063	.00010	495.50	.81916	.000038	.00010
497.00	.81233	.000096	.00010	498.50	.80115	.000044	.00010

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TABLE 4 (cont)

Wave-Length (nm)	T	ΔT Standard Error	ΔT System-atic	Wave-Length (nm)	T	ΔT Standard Error	ΔT System-atic
500.00	.78689	.000060	.00010	501.50	.77050	.000062	.00010
503.00	.75433	.000047	.00010	504.50	.73899	.000046	.00010
506.00	.72185	.000070	.00010	507.50	.69622	.000054	.00010
509.00	.65070	.000060	.00010	510.50	.58889	.000023	.00010
512.00	.53799	.000044	.00010	513.50	.51737	.000050	.00010
515.00	.53523	.000035	.00010	516.50	.56547	.000033	.00010
518.00	.58503	.000042	.00010	519.50	.59603	.000038	.00010
521.00	.57933	.000049	.00010	522.50	.51464	.000058	.00010
524.00	.43678	.000044	.00010	525.50	.40684	.000029	.00010
527.00	.39690	.000057	.00010	528.50	.36734	.000019	.00010
530.00	.36174	.000012	.00010	531.50	.37185	.000046	.00010
533.00	.38958	.000046	.00010	534.50	.45072	.000037	.00010
536.00	.54457	.000052	.00010	537.50	.63283	.000038	.00010
539.00	.69863	.000066	.00010	540.50	.75111	.000028	.00010
542.00	.79247	.000062	.00010	543.50	.82093	.000050	.00010
545.00	.84006	.000043	.00010	546.50	.85304	.000068	.00010
548.00	.86137	.000073	.00010	549.50	.86532	.000043	.00010
551.00	.86587	.000068	.00010	552.50	.86526	.000035	.00010
554.00	.86482	.000057	.00010	555.50	.86353	.000038	.00010
557.00	.85997	.000035	.00010	558.50	.85316	.000088	.00010
560.00	.84237	.000045	.00010	561.50	.82504	.000060	.00010
563.00	.79728	.000048	.00010	564.50	.75134	.000031	.00010
566.00	.66681	.000052	.00010	567.50	.50244	.000030	.00010
569.00	.25719	.000033	.00010	570.50	.07751	.000011	.00010
572.00	.02604	.000010	.00010	573.50	.02536	.000013	.00010
575.00	.03443	.000011	.00010	576.50	.03857	.000013	.00010
578.00	.05117	.000012	.00010	579.50	.06944	.000014	.00010
581.00	.06863	.000007	.00010	582.50	.03836	.000014	.00010
584.00	.01549	.000007	.00010	585.50	.01027	.000006	.00010
587.00	.01577	.000016	.00010	588.50	.02979	.000022	.00010
590.00	.04702	.000023	.00010	591.50	.05748	.000009	.00010
593.00	.07588	.000013	.00010	594.50	.11929	.000028	.00010
596.00	.19036	.000034	.00010	597.50	.26661	.000020	.00010
599.00	.32378	.000038	.00010	600.50	.38146	.000043	.00010
602.00	.44667	.000041	.00010	603.50	.51047	.000043	.00010
605.00	.56569	.000048	.00010	606.50	.60327	.000074	.00010
608.00	.62507	.000047	.00010	609.50	.64527	.000041	.00010
611.00	.68076	.000072	.00010	612.50	.73522	.000054	.00010
614.00	.79774	.000073	.00010	615.50	.84664	.000067	.00010
617.00	.87427	.000086	.00010	618.50	.88467	.000056	.00010

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TABLE 4 (cont)

Wave-Length (nm)	T	ΔT Standard Error	ΔT Systematic	Wave-Length (nm)	T	ΔT Standard Error	ΔT Systematic
620.00	.88413	.000042	.00010	621.50	.87711	.000071	.00010
623.00	.87047	.000037	.00010	624.50	.87083	.000060	.00010
626.00	.87359	.000066	.00010	627.50	.87310	.000069	.00010
629.00	.87008	.000069	.00010	630.50	.87085	.000064	.00010
632.00	.87569	.000048	.00010	633.50	.88221	.000043	.00010
635.00	.88828	.000124	.00010	636.50	.89117	.000089	.00010
638.00	.89322	.000035	.00010	639.50	.89543	.000062	.00010
641.00	.89755	.000085	.00010	642.50	.89905	.000051	.00010
644.00	.90014	.000052	.00010	645.50	.90080	.000043	.00010
647.00	.90124	.000041	.00010	648.50	.90112	.000060	.00010
650.00	.90106	.000034	.00010	651.50	.90149	.000079	.00010
653.00	.90218	.000054	.00010	654.50	.90300	.000066	.00010
656.00	.90301	.000082	.00010	657.50	.90256	.000044	.00010
659.00	.90198	.000049	.00010	660.50	.90115	.000091	.00010
662.00	.89951	.000104	.00010	663.50	.89656	.000071	.00010
665.00	.89155	.000045	.00010	666.50	.88540	.000079	.00010
668.00	.87844	.000079	.00010	669.50	.87177	.000085	.00010
671.00	.86691	.000077	.00010	672.50	.86284	.000056	.00010
674.00	.85883	.000035	.00010	675.50	.85369	.000076	.00010
677.00	.84419	.000077	.00010	678.50	.83115	.000053	.00010
680.00	.81611	.000076	.00010	681.50	.79779	.000063	.00010
683.00	.77773	.000111	.00010	684.50	.76323	.000051	.00010
686.00	.77287	.000111	.00010	687.50	.79255	.000057	.00010
689.00	.80954	.000080	.00010	690.50	.82685	.000045	.00010
692.00	.84399	.000066	.00010	693.50	.85936	.000052	.00010
695.00	.87138	.000058	.00010	696.50	.88070	.000036	.00010
698.00	.88764	.000054	.00010	699.50	.89254	.000029	.00010
701.00	.89577	.000056	.00010	702.50	.89798	.000040	.00010
704.00	.89957	.000049	.00010	705.50	.90089	.000060	.00010
707.00	.90166	.000056	.00010	708.50	.90231	.000071	.00010
710.00	.90225	.000066	.00010	711.50	.90168	.000071	.00010
713.00	.90041	.000056	.00010	714.50	.89850	.000060	.00010
716.00	.89593	.000042	.00010	717.50	.89232	.000040	.00010
719.00	.88755	.000075	.00010	720.50	.88137	.000049	.00010
722.00	.87336	.000061	.00010	723.50	.86256	.000086	.00010
725.00	.84758	.000054	.00010	726.50	.82620	.000070	.00010
728.00	.79437	.000071	.00010	729.50	.74399	.000039	.00010
731.00	.66643	.000039	.00010	732.50	.55165	.000033	.00010
734.00	.40413	.000053	.00010	735.50	.26498	.000040	.00010
737.00	.17772	.000064	.00010	738.50	.14784	.000028	.00010

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TABLE 4 (cont)

Wave-Length (nm)	T	ΔT Standard Error	ΔT Systematic	Wave-Length (nm)	T	ΔT Standard Error	ΔT Systematic
740.00	.14400	.000017	.00010	741.50	.14841	.000015	.00010
743.00	.16928	.000022	.00010	744.50	.19331	.000020	.00010
746.00	.19302	.000027	.00010	747.50	.17683	.000014	.00010
749.00	.17678	.000040	.00010	750.50	.20220	.000026	.00010
752.00	.24335	.000050	.00010	753.50	.29149	.000049	.00010
755.00	.34682	.000095	.00010	756.50	.40869	.000063	.00010
758.00	.47017	.000099	.00010	759.50	.52523	.000164	.00010
761.00	.57256	.000071	.00010	762.50	.61237	.000077	.00010
764.00	.64917	.000089	.00010	765.50	.68412	.000093	.00010
767.00	.71596	.000088	.00010	768.50	.74294	.000080	.00010
770.00	.76342	.000040	.00010	771.50	.77754	.000144	.00010
773.00	.78603	.000082	.00010	774.50	.78934	.000082	.00010
776.00	.78665	.000098	.00010	777.50	.77668	.000050	.00010
779.00	.75800	.000083	.00010	780.50	.73040	.000120	.00010

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U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Director

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Certificate

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The measurements on which these tables are based were made at 25 °C with a high-precision reference spectrophotometer that has a wavelength accuracy of 0.04 nm. Table 3 indicates the estimated random (as obtained from 4 sets of measurements on a single filter) and systematic errors of the transmittance minima given in Table 1. Table 2 also indicates the range of the measured wavelengths of the inflection points. Trial calibrations made on several instruments, using both minima and inflection points, indicate that wavelength corrections made with these SRM's can be accurate to 0.2 nm. The uncertainty of a calibration, however, will depend upon the stability and other characteristics of a particular instrument.

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The spectral transmittance as a function of wavelength for a filter representative of the melt is given in Table 4. These values are not certified but are provided for use as specified in SP 260-66. *They should not be used to check the photometric scale of a spectrophotometer.*

It is recommended that the filter be handled only by its edges and when not in use it should be stored in the box provided. If cleaning is necessary, wet the filter with water and rub gently with optical lens tissue soaked with a mild soap solution, rinse with distilled water, rinse with isoprophyl alcohol, and rinse again with distilled water. Dry after each rinsing by wiping lightly with optical lens tissue.

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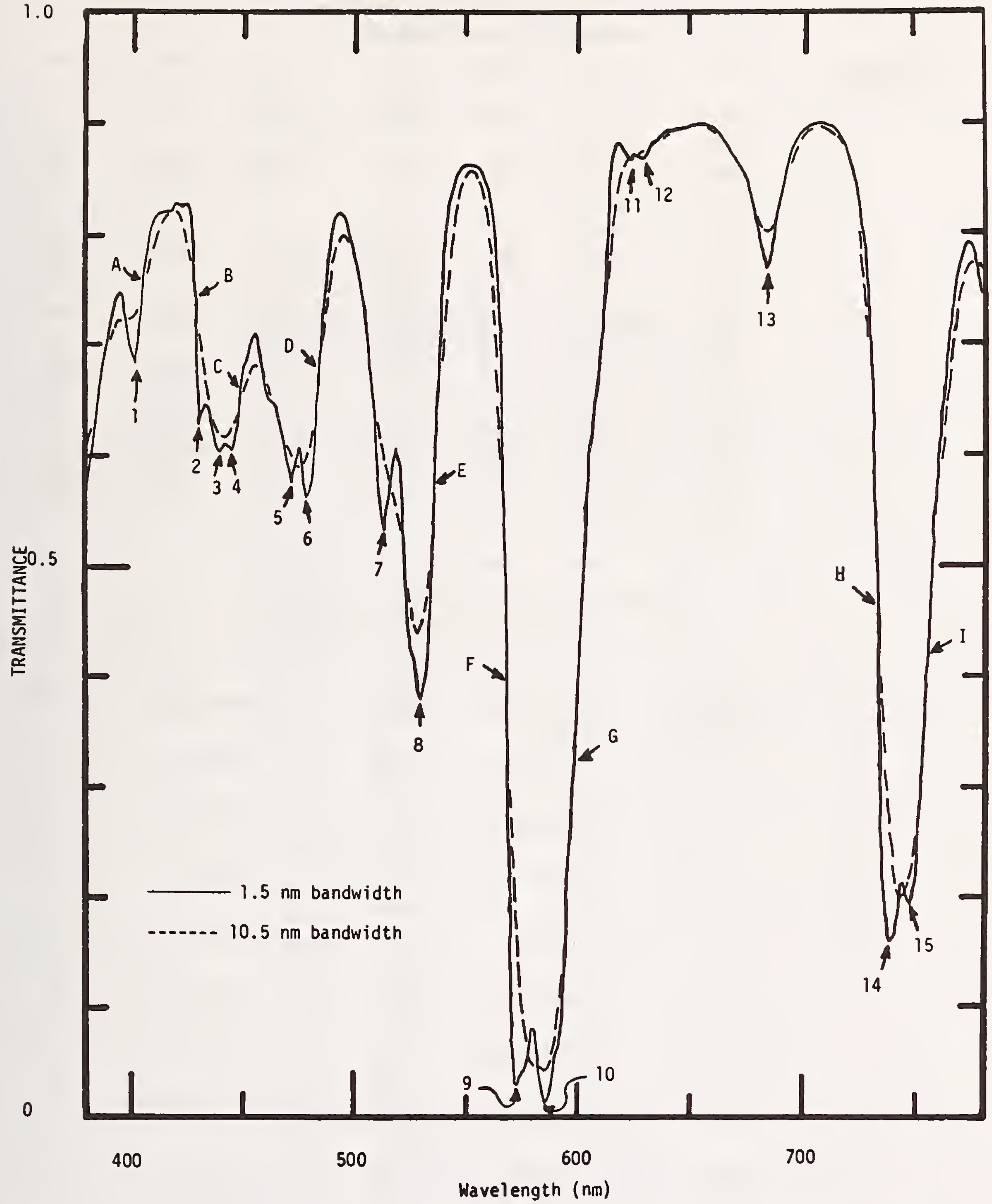
January 8, 1980

(over)

George A. Uriano, Chief
Office of Standard Reference Materials



Spectral transmittance of a typical didymium glass filter. Numbers indicate the principal points of minimum transmittance and letters indicate the principal points of inflection.

Table 1

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Minima for the Indicated Bandwidths

Bandwidth Minimum No.	1.5 nm	3.0 nm	4.5 nm	6.0 nm	7.5 nm	9.0 nm	10.5 nm
1	402.42	401.81	401.69	401.66	401.42	400.95	
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7	513.45	513.61	513.89	514.31	515.38		
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9	572.69	573.27	574.21	575.11	576.59		
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11	623.62	624.02					
12	629.53	629.41	628.56	627.03	627.02		
13	684.66	684.68	684.71	684.72	684.71	684.66	684.58
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15	748.28	748.10					

Table 2

Wavelengths and Transmittances at Nine Selected

Points of Inflection

Point Identification	Wavelength (nm)	Range* (nm)	Transmittance†
A	406.44	+.08 −.06	0.7760
B	429.43	+.05 −.05	.7359
C	449.49	+.06 −.06	.6516
D	484.84	+.10 −.15	.6758
E	536.50	+.06 −.09	.5805
F	568.15	+.08 −.04	.4023
G	599.05	+.05 −.07	.3348
H	733.39	+.06 −.03	.4719
I	756.45	+.01 −.02	.4177

*The range of wavelengths within which the wavelength for the given transmittance will fall for symmetric triangular passbands with half-height bandwidths from 1.5 to 10.5 nm.

†These values of transmittance are not certified.

Table 3

Estimated Random and Systematic Errors of the Transmittance Minima

Band Number	Nominal Wavelength of Minimum Transmittance	Standard Deviation for Indicated Bandwidth						
		1.5 nm	3.0 nm	4.5 nm	6.0 nm	7.5 nm	9.0 nm	10.5 nm
1	402 nm	0.015 nm (0.07)†	0.018 nm	0.020 nm	0.021 nm	0.025 nm	0.029 nm	–
2	431	0.013 (0.06)	0.010	–	–	–	–	–
3	440	0.021 (0.05)	0.013	0.050	0.028	0.009	0.005	0.007 nm (0.25)
4	446	0.023 (0.06)	0.029	–	–	–	–	–
5	473	0.011 (0.08)	0.012	0.032	–	–	–	–
6	479	0.015 (0.15)	0.013	0.009	0.009	0.009	0.014	0.017 (0.25)
7	573	0.022 (0.08)	0.016	0.014	0.013	0.010	–	–
8	530	0.012 (0.21)	0.010	0.010	0.011	0.010	0.011	0.010 (0.25)
9	573	0.004 (0.05)	0.010	0.010	0.012	0.014	–	–
10	585	0.007 (0.06)	0.004	0.007	0.008	0.008	0.007	0.007 (0.25)
11	624	0.058 (0.06)	0.061	–	–	–	–	–
12	630	0.210 (0.02)	0.120	0.171	0.133	0.091	–	–
13	685	0.019 (0.05)	0.029	0.024	0.017	0.014	0.014	0.012 (0.25)
14	740	0.009 (0.25)	0.013	0.011	0.010	0.009	0.009	0.010 (0.25)
15	748	0.020 (0.04)	0.016	–	–	–	–	–

†Values in parentheses are estimates of the systematic error. The estimates for the 1.5 nm bandwidth were obtained from the data taken at 1.5 nm intervals as compared to data taken at 0.15 nm intervals. The method of estimating the systematic errors for the 10.5 nm bandwidth is described in Section 3.4 in SP 260-66.

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TABLE 4

Transmittance (T) as a function of wavelength from 380 to 780.5 nm for a bandwidth of 1.5 nm. The estimates of random and systematic errors are also provided. These data are not certified.

Wave-Length (nm)	T	ΔT Standard Error	ΔT Systematic	Wave-Length (nm)	T	ΔT Standard Error	ΔT Systematic
380.00	.61832	.000054	.00010	381.50	.63896	.000085	.00010
383.00	.66210	.000064	.00010	384.50	.68291	.000079	.00010
386.00	.70112	.000047	.00010	387.50	.71885	.000071	.00010
389.00	.73590	.000095	.00010	390.50	.74954	.000040	.00010
392.00	.76201	.000083	.00010	393.50	.77104	.000055	.00010
395.00	.77620	.000064	.00010	396.50	.77123	.000038	.00010
398.00	.75013	.000069	.00010	399.50	.72419	.000079	.00010
401.00	.70966	.000070	.00010	402.50	.69505	.000071	.00010
404.00	.73683	.000057	.00010	405.50	.75886	.000047	.00010
407.00	.77756	.000088	.00010	408.50	.80673	.000072	.00010
410.00	.82277	.000058	.00010	411.50	.82813	.000038	.00010
413.00	.83115	.000060	.00010	414.50	.83287	.000075	.00010
416.00	.83222	.000060	.00010	417.50	.83200	.000029	.00010
419.00	.83277	.000059	.00010	420.50	.83844	.000066	.00010
422.00	.83711	.000064	.00010	423.50	.83328	.000052	.00010
425.00	.83511	.000049	.00010	426.50	.83414	.000045	.00010
428.00	.81620	.000055	.00010	429.50	.72877	.000023	.00010
431.00	.62212	.000096	.00010	432.50	.63478	.000039	.00010
434.00	.64183	.000057	.00010	435.50	.63392	.000059	.00010
437.00	.61858	.000049	.00010	438.50	.60090	.000038	.00010
440.00	.59163	.000064	.00010	441.50	.59496	.000053	.00010
443.00	.60082	.000063	.00010	444.50	.59576	.000068	.00010
446.00	.59315	.000055	.00010	447.50	.60769	.000044	.00010
449.00	.63551	.000083	.00010	450.50	.66313	.000060	.00010
452.00	.68002	.000069	.00010	453.50	.69110	.000057	.00010
455.00	.70206	.000027	.00010	456.50	.70341	.000033	.00010
458.00	.69051	.000025	.00010	459.50	.66807	.000049	.00010
461.00	.64890	.000057	.00010	462.50	.63966	.000060	.00010
464.00	.63837	.000070	.00010	465.50	.63353	.000040	.00010
467.00	.62094	.000072	.00010	468.50	.60572	.000053	.00010
470.00	.59042	.000008	.00010	471.50	.57213	.000072	.00010
473.00	.56160	.000033	.00010	474.50	.58414	.000031	.00010
476.00	.59528	.000052	.00010	477.50	.56003	.000052	.00010
479.00	.54953	.000070	.00010	480.50	.55659	.000036	.00010
482.00	.57326	.000025	.00010	483.50	.62009	.000040	.00010
485.00	.67764	.000052	.00010	486.50	.72126	.000072	.00010
488.00	.75412	.000045	.00010	489.50	.78321	.000082	.00010
491.00	.80556	.000035	.00010	492.50	.81759	.000060	.00010
494.00	.82117	.000063	.00010	495.50	.81916	.000038	.00010
497.00	.81233	.000096	.00010	498.50	.80115	.000044	.00010

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TABLE 4 (cont)

Wave-Length (nm)	T	ΔT Standard Error	ΔT System-atic	Wave-Length (nm)	T	ΔT Standard Error	ΔT System-atic
500.00	.78689	.000060	.00010	501.50	.77050	.000062	.00010
503.00	.75433	.000047	.00010	504.50	.73899	.000046	.00010
506.00	.72185	.000070	.00010	507.50	.69622	.000054	.00010
509.00	.65070	.000060	.00010	510.50	.58889	.000023	.00010
512.00	.53799	.000044	.00010	513.50	.51737	.000050	.00010
515.00	.53523	.000035	.00010	516.50	.56547	.000033	.00010
518.00	.58503	.000042	.00010	519.50	.59603	.000038	.00010
521.00	.57933	.000049	.00010	522.50	.51464	.000058	.00010
524.00	.43678	.000044	.00010	525.50	.40684	.000029	.00010
527.00	.39690	.000057	.00010	528.50	.36734	.000019	.00010
530.00	.36174	.000012	.00010	531.50	.37185	.000046	.00010
533.00	.38958	.000046	.00010	534.50	.45072	.000037	.00010
536.00	.54457	.000052	.00010	537.50	.63283	.000038	.00010
539.00	.69863	.000066	.00010	540.50	.75111	.000028	.00010
542.00	.79247	.000062	.00010	543.50	.82093	.000050	.00010
545.00	.84006	.000043	.00010	546.50	.85304	.000068	.00010
548.00	.86137	.000073	.00010	549.50	.86532	.000043	.00010
551.00	.86587	.000068	.00010	552.50	.86526	.000035	.00010
554.00	.86482	.000057	.00010	555.50	.86353	.000038	.00010
557.00	.85997	.000035	.00010	558.50	.85316	.000088	.00010
560.00	.84237	.000045	.00010	561.50	.82504	.000060	.00010
563.00	.79728	.000048	.00010	564.50	.75134	.000031	.00010
566.00	.66681	.000052	.00010	567.50	.50244	.000030	.00010
569.00	.25719	.000033	.00010	570.50	.07751	.000011	.00010
572.00	.02604	.000010	.00010	573.50	.02536	.000013	.00010
575.00	.03443	.000011	.00010	576.50	.03857	.000013	.00010
578.00	.05117	.000012	.00010	579.50	.06944	.000014	.00010
581.00	.06863	.000007	.00010	582.50	.03836	.000014	.00010
584.00	.01549	.000007	.00010	585.50	.01027	.000006	.00010
587.00	.01577	.000016	.00010	588.50	.02979	.000022	.00010
590.00	.04702	.000023	.00010	591.50	.05748	.000009	.00010
593.00	.07588	.000013	.00010	594.50	.11929	.000028	.00010
596.00	.19036	.000034	.00010	597.50	.26661	.000020	.00010
599.00	.32378	.000038	.00010	600.50	.38146	.000043	.00010
602.00	.44667	.000041	.00010	603.50	.51047	.000043	.00010
605.00	.56569	.000048	.00010	606.50	.60327	.000074	.00010
608.00	.62507	.000047	.00010	609.50	.64527	.000041	.00010
611.00	.68076	.000072	.00010	612.50	.73522	.000054	.00010
614.00	.79774	.000073	.00010	615.50	.84664	.000067	.00010
617.00	.87427	.000086	.00010	618.50	.88467	.000056	.00010

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TABLE 4 (cont)

Wave-Length (nm)	T	ΔT Standard Error	ΔT Systematic	Wave-Length (nm)	T	ΔT Standard Error	ΔT Systematic
620.00	.88413	.000042	.00010	621.50	.87711	.000071	.00010
623.00	.87047	.000037	.00010	624.50	.87083	.000060	.00010
626.00	.87359	.000066	.00010	627.50	.87310	.000069	.00010
629.00	.87008	.000069	.00010	630.50	.87085	.000064	.00010
632.00	.87569	.000048	.00010	633.50	.88221	.000043	.00010
635.00	.88828	.000124	.00010	636.50	.89117	.000089	.00010
638.00	.89322	.000035	.00010	639.50	.89543	.000062	.00010
641.00	.89755	.000085	.00010	642.50	.89905	.000051	.00010
644.00	.90014	.000052	.00010	645.50	.90080	.000043	.00010
647.00	.90124	.000041	.00010	648.50	.90112	.000060	.00010
650.00	.90106	.000034	.00010	651.50	.90149	.000079	.00010
653.00	.90218	.000054	.00010	654.50	.90300	.000066	.00010
656.00	.90301	.000082	.00010	657.50	.90256	.000044	.00010
659.00	.90198	.000049	.00010	660.50	.90115	.000091	.00010
662.00	.89951	.000104	.00010	663.50	.89656	.000071	.00010
665.00	.89155	.000045	.00010	666.50	.88540	.000079	.00010
668.00	.87844	.000079	.00010	669.50	.87177	.000085	.00010
671.00	.86691	.000077	.00010	672.50	.86284	.000056	.00010
674.00	.85883	.000035	.00010	675.50	.85369	.000076	.00010
677.00	.84419	.000077	.00010	678.50	.83115	.000053	.00010
680.00	.81611	.000076	.00010	681.50	.79779	.000063	.00010
683.00	.77773	.000111	.00010	684.50	.76323	.000051	.00010
686.00	.77287	.000111	.00010	687.50	.79255	.000057	.00010
689.00	.80954	.000080	.00010	690.50	.82685	.000045	.00010
692.00	.84399	.000066	.00010	693.50	.85936	.000052	.00010
695.00	.87138	.000058	.00010	696.50	.88070	.000036	.00010
698.00	.88764	.000054	.00010	699.50	.89254	.000029	.00010
701.00	.89577	.000056	.00010	702.50	.89798	.000040	.00010
704.00	.89957	.000049	.00010	705.50	.90039	.000060	.00010
707.00	.90166	.000056	.00010	708.50	.90231	.000071	.00010
710.00	.90225	.000066	.00010	711.50	.90168	.000071	.00010
713.00	.90041	.000056	.00010	714.50	.89850	.000060	.00010
716.00	.89593	.000042	.00010	717.50	.89232	.000040	.00010
719.00	.88755	.000075	.00010	720.50	.88137	.000049	.00010
722.00	.87336	.000061	.00010	723.50	.86256	.000086	.00010
725.00	.84758	.000054	.00010	726.50	.82620	.000070	.00010
728.00	.79437	.000071	.00010	729.50	.74399	.000039	.00010
731.00	.66643	.000039	.00010	732.50	.55165	.000033	.00010
734.00	.40413	.000053	.00010	735.50	.26498	.000040	.00010
737.00	.17772	.000064	.00010	738.50	.14784	.000028	.00010

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TABLE 4 (cont)

Wave-Length (nm)	T	ΔT Standard Error	ΔT Systematic	Wave-Length (nm)	T	ΔT Standard Error	ΔT Systematic
740.00	.14400	.000017	.00010	741.50	.14841	.000015	.00010
743.00	.16928	.000022	.00010	744.50	.19331	.000020	.00010
746.00	.19302	.000027	.00010	747.50	.17683	.000014	.00010
749.00	.17678	.000040	.00010	750.50	.20220	.000026	.00010
752.00	.24335	.000050	.00010	753.50	.29149	.000049	.00010
755.00	.34682	.000095	.00010	756.50	.40869	.000063	.00010
758.00	.47017	.000099	.00010	759.50	.52523	.000164	.00010
761.00	.57256	.000071	.00010	762.50	.61237	.000077	.00010
764.00	.64917	.000089	.00010	765.50	.68412	.000093	.00010
767.00	.71596	.000088	.00010	768.50	.74294	.000080	.00010
770.00	.76342	.000040	.00010	771.50	.77754	.000144	.00010
773.00	.78603	.000082	.00010	774.50	.78934	.000082	.00010
776.00	.78665	.000098	.00010	777.50	.77668	.000050	.00010
779.00	.75800	.000083	.00010	780.50	.73040	.000120	.00010

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U.S. Department of Commerce
Juanita M. Kreps
Secretary
National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards
Certificate

Standard Reference Material 2013

Didymium Glass Filter for Checking the Wavelength Scale of Spectrophotometers

Kenneth L. Eckerle and William H. Venable, Jr.

Serial Number:

This SRM is intended for use in calibrating the wavelength scale in the visible wavelength region of scanning spectrophotometers having nominal bandwidths in the range 1.5 to 10.5 nm. Depending upon the bandwidth of the spectrophotometer, anywhere from 14 to 24 wavelength corrections can be determined from 400 to 760 nm. Detailed instructions on the use of this SRM and examples of its use are given in NBS Special Publication 260-66. Each didymium-glass filter is identified by the SRM number and a serial number.

The wavelengths of the transmittance minima as obtained from measurements on each filter are given in Table 1. These values are given for seven equally spaced values of the half-height width of triangular passbands. The minima number is identified in the figure that illustrates the spectral transmittance as a function of wavelength. The wavelength values of nine points of inflection on the spectral transmittance curve as obtained on three filters are given in Table 2. These inflection points are representative of the melt and are also identified in the figure. These inflection points should only be used with the transmittance minima as described in Sections 2.2 and 2.3 in SP 260-66.

The measurements on which these tables are based were made at 25 °C with a high-precision reference spectrophotometer that has a wavelength accuracy of 0.04 nm. Table 3 indicates the estimated random (as obtained from 4 sets of measurements on a single filter) and systematic errors of the transmittance minima given in Table 1. Table 2 also indicates the range of the measured wavelengths of the inflection points. Trial calibrations made on several instruments, using both minima and inflection points, indicate that wavelength corrections made with these SRM's can be accurate to 0.2 nm. The uncertainty of a calibration, however, will depend upon the stability and other characteristics of a particular instrument.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Office of Standard Reference Materials by R. K. Kirby.

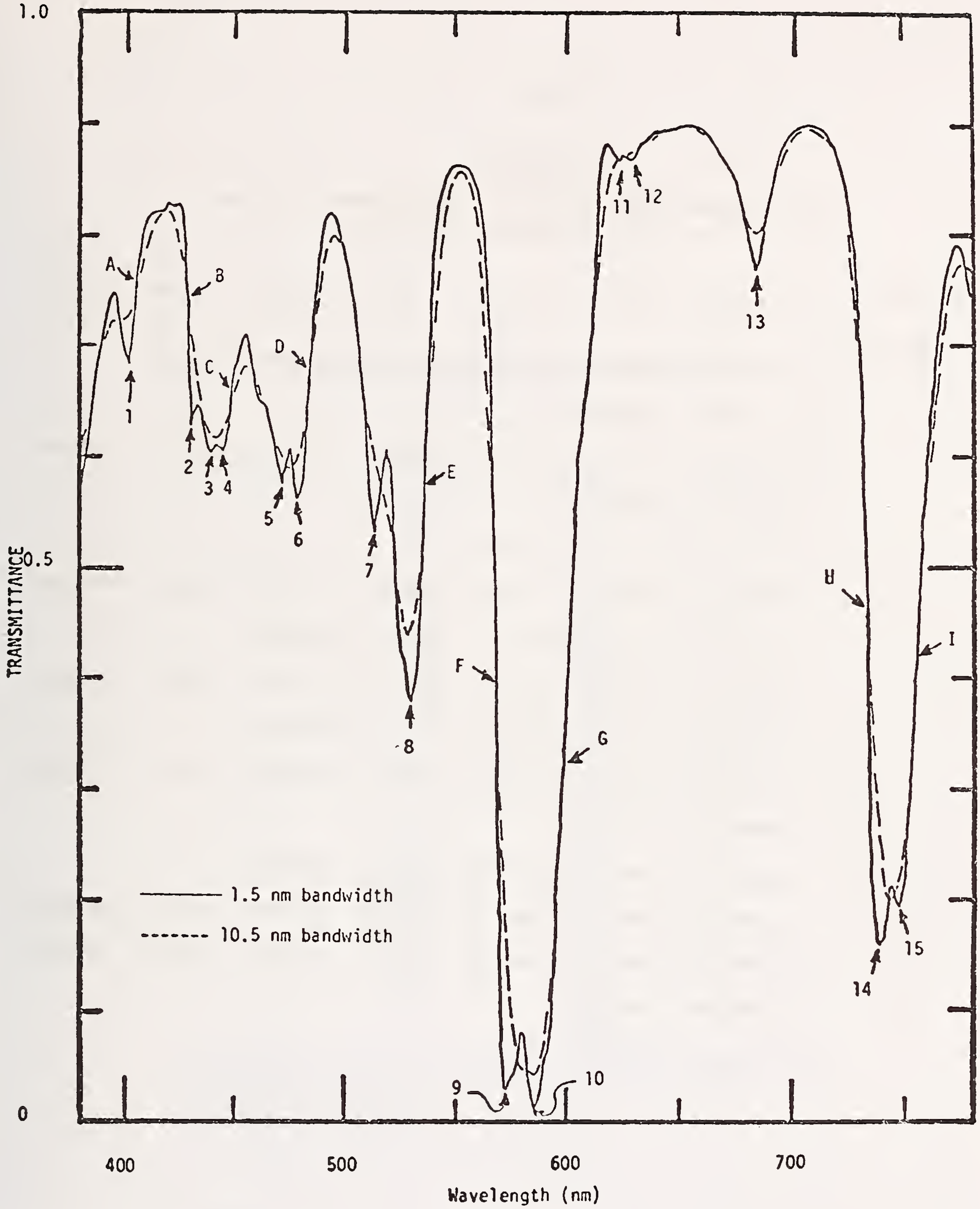
The spectral transmittance as a function of wavelength for this filter is given in Table 4. These values are not certified but are provided for use as specified in SP 260-66. *They should not be used to check the photometric scale of a spectrophotometer.*

It is recommended that the filter be handled only by its edges and when not in use it should be stored in the box provided. If cleaning is necessary, wet the filter with water and rub gently with optical lens tissue soaked with a mild soap solution, rinse with distilled water, rinse with isopropyl alcohol, and rinse again with distilled water. Dry after each rinsing by wiping lightly with optical lens tissue.

Washington, D.C. 20234
January 8, 1980

(over)

George A. Uriano, Chief
Office of Standard Reference Materials



Spectral transmittance of a typical didymium glass filter. Numbers indicate the principal points of minimum transmittance and letters indicate the principal points of inflection.

TABLE 1

SRM 2013
FILTER F1
Wavelengths (nm) of the transmittance minima
for the indicated bandwidths.

BANDWIDTH / MINIMUM NO.	1.5NM	3.0NM	4.5NM	6.0NM	7.5NM	9.0NM	10.5NM
1	402.37	401.69	401.50	401.37	400.99	400.24	
2	431.51	432.46					
3	440.20	440.41	441.62	442.43	442.26	441.92	441.06
4	445.59	445.17					
5	472.73	472.60	472.97				
6	478.81	479.28	479.22	478.28	477.37	476.54	475.68
7	513.43	513.61	513.89	514.31	515.38		
8	529.65	530.11	530.00	529.54	529.32	529.17	528.92
9	572.68	573.25	574.17	575.06	576.51		
10	585.35	585.54	585.76	586.02	585.99	585.36	584.44
11	623.58	623.96					
12	629.39	629.28	628.39	627.04	626.98		
13	684.62	684.67	684.72	684.74	684.72	684.68	684.59
14	739.87	739.95	740.25	740.91	742.00	742.95	743.63
15	748.25	748.09					

Table 2

Wavelengths and Transmittances at Nine Selected Points of Inflection

Point Identification	Wavelength (nm)	Range* (nm)	Transmittance†
A	406.38	+.08 −.06	0.7562
B	429.42	+.05 −.05	.7293
C	449.45	+.06 −.06	.6514
D	484.77	+.10 −.15	.6743
E	536.52	+.06 −.09	.5801
F	568.16	+.08 −.04	.4008
G	598.99	+.05 −.07	.3346
H	733.45	+.06 −.03	.4730
I	756.48	+.01 −.02	.4215

*The range of wavelengths within which the wavelength for the given transmittance will fall for symmetric triangular passbands with half-height bandwidths from 1.5 to 10.5 nm.

†These values of transmittance are not certified.

SRM 2013

Table 3

Estimated Random and Systematic Errors of the Transmittance Minima

Band Number	Nominal Wavelength of Minimum Transmittance	Standard Deviation for Indicated Bandwidth						
		1.5 nm	3.0 nm	4.5 nm	6.0 nm	7.5 nm	9.0 nm	10.5 nm
1	402 nm	0.015 nm (0.07)†	0.018 nm	0.020 nm	0.021 nm	0.025 nm	0.029 nm	–
2	431	0.013 (0.06)	0.010	–	–	–	–	–
3	440	0.021 (0.05)	0.013	0.050	0.028	0.009	0.005	0.007 nm (0.25)
4	446	0.023 (0.06)	0.029	–	–	–	–	–
5	473	0.011 (0.08)	0.012	0.032	–	–	–	–
6	479	0.015 (0.15)	0.013	0.009	0.009	0.009	0.014	0.017 (0.25)
7	513 ~~573~~	0.022 (0.08)	0.016	0.014	0.013	0.010	–	–
8	530	0.012 (0.21)	0.010	0.010	0.011	0.010	0.011	0.010 (0.25)
9	573	0.004 (0.05)	0.010	0.010	0.012	0.014	–	–
10	585	0.007 (0.06)	0.004	0.007	0.008	0.008	0.007	0.007 (0.25)
11	624	0.058 (0.06)	0.061	–	–	–	–	–
12	630	0.210 (0.02)	0.120	0.171	0.133	0.091	–	–
13	685	0.019 (0.05)	0.029	0.024	0.017	0.014	0.014	0.012 (0.25)
14	740	0.009 (0.25)	0.013	0.011	0.010	0.009	0.009	0.010 (0.25)
15	748	0.020 (0.04)	0.016	–	–	–	–	–

†Values in parentheses are estimates of the systematic error. The estimates for the 1.5 nm bandwidth were obtained from the data taken at 1.5 nm intervals as compared to data taken at 0.15 nm intervals. The method of estimating the systematic errors for the 10.5 nm bandwidth is described in Section 3.4 in SP 260-66.

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TABLE 4

Transmittance (T) as a function of wavelength from 380 to 780.5 nm for a bandwidth of 1.5 nm. The estimates of random and systematic errors are also provided. These data are not certified.

SRM 2013
FILTER F1

WAVE-LENGTH (NM)	T	ΔT STANDARD ERROR	ΔT SYSTEM-ATIC	WAVE-LENGTH (NM)	T	ΔT STANDARD ERROR	ΔT SYSTEM-ATIC
380.00	.56826	.000170	.00020	381.50	.59269	.000157	.00020
383.00	.61819	.000414	.00020	384.50	.64237	.000146	.00020
386.00	.66334	.000164	.00020	387.50	.68330	.000040	.00020
389.00	.70209	.000180	.00020	390.50	.71751	.000170	.00020
392.00	.73218	.000311	.00020	393.50	.74297	.000285	.00020
395.00	.75013	.000192	.00020	396.50	.74790	.000287	.00020
398.00	.73081	.000117	.00020	399.50	.70879	.000257	.00020
401.00	.69713	.000221	.00020	402.50	.68509	.000284	.00020
404.00	.72530	.000145	.00020	405.50	.74736	.000149	.00020
407.00	.76637	.000251	.00020	408.50	.79408	.000298	.00020
410.00	.80982	.000363	.00020	411.50	.81581	.000324	.00020
413.00	.81971	.000283	.00020	414.50	.82266	.000274	.00020
416.00	.82240	.000759	.00020	417.50	.82338	.000166	.00020
419.00	.82486	.000104	.00020	420.50	.83065	.000296	.00020
422.00	.83008	.000438	.00020	423.50	.82672	.000155	.00020
425.00	.82968	.000319	.00020	426.50	.82907	.000235	.00020
428.00	.81277	.000419	.00020	429.50	.73163	.000432	.00020
431.00	.63172	.000282	.00020	432.50	.64288	.000177	.00020
434.00	.65066	.000080	.00020	435.50	.64411	.000700	.00020
437.00	.63043	.000157	.00020	438.50	.61489	.000182	.00020
440.00	.60685	.000107	.00020	441.50	.61053	.000129	.00020
443.00	.61614	.000432	.00020	444.50	.61099	.000079	.00020
446.00	.60856	.000158	.00020	447.50	.62206	.000281	.00020
449.00	.64896	.000076	.00020	450.50	.67502	.000303	.00020
452.00	.69049	.000149	.00020	453.50	.70122	.000186	.00020
455.00	.71162	.000204	.00020	456.50	.71301	.000432	.00020
458.00	.70059	.000321	.00020	459.50	.67909	.000086	.00020
461.00	.66115	.000298	.00020	462.50	.65213	.000162	.00020
464.00	.65116	.000283	.00020	465.50	.64672	.000567	.00020
467.00	.63481	.000079	.00020	468.50	.62065	.000153	.00020
470.00	.60647	.000136	.00020	471.50	.58911	.000250	.00020
473.00	.57923	.000046	.00020	474.50	.59973	.000142	.00020
476.00	.61005	.000147	.00020	477.50	.57523	.000162	.00020
479.00	.56568	.000153	.00020	480.50	.57355	.000152	.00020
482.00	.59022	.000046	.00020	483.50	.63493	.000428	.00020
485.00	.68932	.000182	.00020	486.50	.72979	.000150	.00020
488.00	.76066	.000155	.00020	489.50	.78717	.000195	.00020
491.00	.80852	.000562	.00020	492.50	.81985	.000123	.00020
494.00	.82309	.000165	.00020	495.50	.82132	.000217	.00020
497.00	.81518	.000217	.00020	498.50	.80472	.000287	.00020

TABLE 4 (cont)

SRM 2013
FILTER F1

WAVE-LENGTH (NM)	T	ΔT STANDARD ERROR	ΔT SYSTEM-ATIC	WAVE-LENGTH (NM)	T	ΔT STANDARD ERROR	ΔT SYSTEM-ATIC
500.00	.79212	.000040	.00020	501.50	.77617	.000418	.00020
503.00	.76184	.000177	.00020	504.50	.74729	.000448	.00020
506.00	.73157	.000290	.00020	507.50	.70732	.000150	.00020
509.00	.66454	.000053	.00020	510.50	.60528	.000132	.00020
512.00	.55721	.000158	.00020	513.50	.53711	.000219	.00020
515.00	.55442	.000278	.00020	516.50	.58295	.000295	.00020
518.00	.60182	.000176	.00020	519.50	.61259	.000049	.00020
521.00	.59589	.000325	.00020	522.50	.53474	.000066	.00020
524.00	.45922	.000156	.00020	525.50	.43025	.000068	.00020
527.00	.42096	.000052	.00020	528.50	.39112	.000298	.00020
530.00	.38444	.000291	.00020	531.50	.39373	.000133	.00020
533.00	.40937	.000046	.00020	534.50	.46919	.000285	.00020
536.00	.56132	.000291	.00020	537.50	.64638	.000261	.00020
539.00	.70929	.000179	.00020	540.50	.75823	.000230	.00020
542.00	.79724	.000249	.00020	543.50	.82355	.000459	.00020
545.00	.84153	.000133	.00020	546.50	.85337	.000569	.00020
548.00	.86176	.000433	.00020	549.50	.86480	.000114	.00020
551.00	.86603	.000197	.00020	552.50	.86524	.000154	.00020
554.00	.86411	.000248	.00020	555.50	.86393	.000204	.00020
557.00	.86019	.000330	.00020	558.50	.85370	.000415	.00020
560.00	.84381	.000175	.00020	561.50	.82721	.000163	.00020
563.00	.80172	.000166	.00020	564.50	.75799	.000319	.00020
566.00	.67701	.000567	.00020	567.50	.51959	.000042	.00020
569.00	.27505	.000025	.00020	570.50	.08832	.000015	.00020
572.00	.03214	.000140	.00020	573.50	.03157	.000004	.00020
575.00	.04230	.000007	.00020	576.50	.04757	.000140	.00020
578.00	.06218	.000009	.00020	579.50	.08283	.000140	.00020
581.00	.08243	.000013	.00020	582.50	.04771	.000014	.00020
584.00	.02048	.000140	.00020	585.50	.01397	.000140	.00020
587.00	.02077	.000133	.00020	588.50	.03775	.000140	.00020
590.00	.05781	.000281	.00020	591.50	.06923	.000148	.00020
593.00	.08936	.000140	.00020	594.50	.13635	.000025	.00020
596.00	.21181	.000135	.00020	597.50	.29016	.000281	.00020
599.00	.34754	.000141	.00020	600.50	.40480	.000428	.00020
602.00	.46864	.000091	.00020	603.50	.53042	.000211	.00020
605.00	.58254	.000306	.00020	606.50	.61750	.000446	.00020
608.00	.63714	.000224	.00020	609.50	.65620	.000179	.00020
611.00	.68948	.000223	.00020	612.50	.74227	.000301	.00020
614.00	.80146	.000361	.00020	615.50	.84857	.000309	.00020
617.00	.87426	.000174	.00020	618.50	.88386	.000280	.00020

TABLE 4 (cont)

SRM 2013
FILTER F1

WAVE-LENGTH (NM)	T	ΔT STANDARD ERROR	ΔT SYSTEM-ATIC	WAVE-LENGTH (NM)	T	ΔT STANDARD ERROR	ΔT SYSTEM-ATIC
620.00	.88356	.000451	.00020	621.50	.87770	.000300	.00020
623.00	.87126	.000325	.00020	624.50	.87187	.000288	.00020
626.00	.87467	.000305	.00020	627.50	.87343	.000175	.00020
629.00	.87135	.000460	.00020	630.50	.87220	.000318	.00020
632.00	.87635	.000314	.00020	633.50	.88260	.000484	.00020
635.00	.88866	.000442	.00020	636.50	.89092	.000321	.00020
638.00	.89261	.000454	.00020	639.50	.89402	.000429	.00020
641.00	.89653	.000436	.00020	642.50	.89780	.000162	.00020
644.00	.89874	.000301	.00020	645.50	.89908	.000288	.00020
647.00	.89973	.000443	.00020	648.50	.89997	.000153	.00020
650.00	.89978	.000599	.00020	651.50	.89990	.000169	.00020
653.00	.90073	.000444	.00020	654.50	.90233	.000301	.00020
656.00	.90125	.000286	.00020	657.50	.90092	.000151	.00020
659.00	.90117	.000230	.00020	660.50	.90041	.000141	.00020
662.00	.89832	.000319	.00020	663.50	.89540	.000108	.00020
665.00	.89067	.000202	.00020	666.50	.88514	.000196	.00020
668.00	.87868	.000244	.00020	669.50	.87297	.000146	.00020
671.00	.86809	.000126	.00020	672.50	.86472	.000277	.00020
674.00	.86099	.000068	.00020	675.50	.85638	.000177	.00020
677.00	.84756	.000040	.00020	678.50	.83585	.000148	.00020
680.00	.82177	.000569	.00020	681.50	.80482	.000565	.00020
683.00	.78521	.000433	.00020	684.50	.77166	.000701	.00020
686.00	.78125	.000450	.00020	687.50	.79876	.000447	.00020
689.00	.81476	.000446	.00020	690.50	.83135	.000534	.00020
692.00	.84711	.000311	.00020	693.50	.86183	.000173	.00020
695.00	.87321	.000479	.00020	696.50	.88055	.000477	.00020
698.00	.88810	.000139	.00020	699.50	.89189	.000726	.00020
701.00	.89477	.000566	.00020	702.50	.89720	.000450	.00020
704.00	.89838	.000493	.00020	705.50	.89937	.000201	.00020
707.00	.90040	.000699	.00020	708.50	.90159	.000420	.00020
710.00	.90163	.000718	.00020	711.50	.90017	.000256	.00020
713.00	.89930	.000531	.00020	714.50	.89791	.000350	.00020
716.00	.89503	.000235	.00020	717.50	.89179	.000622	.00020
719.00	.88721	.000492	.00020	720.50	.88172	.000751	.00020
722.00	.87433	.000460	.00020	723.50	.86430	.000694	.00020
725.00	.85137	.000694	.00020	726.50	.83115	.000708	.00020
728.00	.80108	.000141	.00020	729.50	.75514	.000303	.00020
731.00	.68274	.000730	.00020	732.50	.57430	.000303	.00020
734.00	.43291	.000088	.00020	735.50	.29391	.000010	.00020
737.00	.20138	.000071	.00020	738.50	.16904	.000011	.00020

TABLE 4 (cont)

SRM 2013
FILTER F1

WAVE-LENGTH (NM)	T	ΔT STANDARD ERROR	ΔT SYSTEM-ATIC	WAVE-LENGTH (NM)	T	ΔT STANDARD ERROR	ΔT SYSTEM-ATIC
740.00	.16460	.000141	.00020	741.50	.16966	.000142	.00020
743.00	.19210	.000150	.00020	744.50	.21749	.000133	.00020
746.00	.21796	.000292	.00020	747.50	.20096	.000137	.00020
749.00	.20125	.000424	.00020	750.50	.22781	.000040	.00020
752.00	.26948	.000566	.00020	753.50	.31813	.000429	.00020
755.00	.37350	.000060	.00020	756.50	.43373	.000428	.00020
758.00	.49359	.000309	.00020	759.50	.54744	.000327	.00020
761.00	.59259	.000129	.00020	762.50	.63097	.000265	.00020
764.00	.66575	.001325	.00020	765.50	.69808	.000720	.00020
767.00	.72868	.000358	.00020	768.50	.75426	.000255	.00020
770.00	.77239	.000468	.00020	771.50	.78515	.001001	.00020
773.00	.79374	.000564	.00020	774.50	.79578	.000900	.00020
776.00	.79383	.000269	.00020	777.50	.78466	.000367	.00020
779.00	.76677	.001005	.00020	780.50	.74176	.000789	.00020

U.S. Department of Commerce
Juanita M. Kreps
Secretary
National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 2014

Didymium Glass Filter for Checking the Wavelength Scale of Spectrophotometers

Kenneth L. Eckerle and William H. Venable, Jr.

Serial Number:

This SRM is intended for use in calibrating the wavelength scale in the visible wavelength region of scanning spectrophotometers having nominal bandwidths in the range 1.5 to 10.5 nm. Depending upon the bandwidth of the spectrophotometer, anywhere from 14 to 24 wavelength corrections can be determined from 400 to 760 nm. Detailed instructions on the use of this SRM and examples of its use are given in NBS Special Publication 260-66. Each didymium-glass filter is identified by the SRM number and a serial number.

The wavelengths of the transmittance minima as obtained from measurements on each filter are given in Table 1. These values are given for seven equally spaced values of the half-height width of triangular passbands. The minima number is identified in the figure that illustrates the spectral transmittance as a function of wavelength. The wavelength values of nine points of inflection on the spectral transmittance curve as obtained on three filters are given in Table 2. These inflection points are representative of the melt and are also identified in the figure. These inflection points should only be used with the transmittance minima as described in Sections 2.2 and 2.3 in SP 260-66.

The measurements on which these tables are based were made at 25 °C with a high-precision reference spectrophotometer that has a wavelength accuracy of 0.04 nm. Table 3 indicates the estimated random (as obtained from 4 sets of measurements on a single filter) and systematic errors of the transmittance minima given in Table 1. Table 2 also indicates the range of the measured wavelengths of the inflection points. Trial calibrations made on several instruments, using both minima and inflection points, indicate that wavelength corrections made with these SRM's can be accurate to 0.2 nm. The uncertainty of a calibration, however, will depend upon the stability and other characteristics of a particular instrument.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Office of Standard Reference Materials by R. K. Kirby.

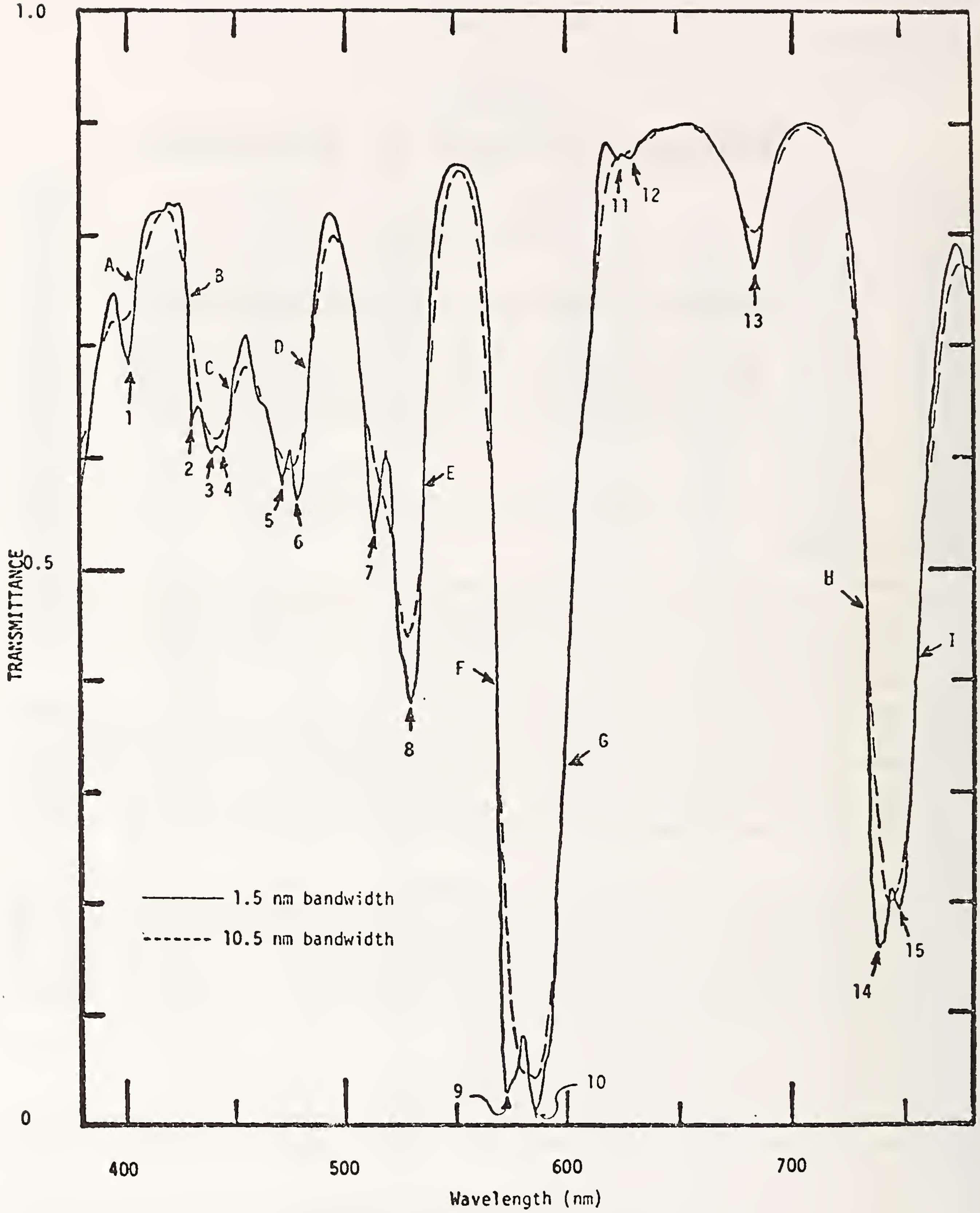
The spectral transmittance as a function of wavelength for this filter is given in Table 4. These values are not certified but are provided for use as specified in SP 260-66. *They should not be used to check the photometric scale of a spectrophotometer.*

It is recommended that the filter be handled only by its edges and when not in use it should be stored in the box provided. If cleaning is necessary, wet the filter with water and rub gently with optical lens tissue soaked with a mild soap solution, rinse with distilled water, rinse with isopropyl alcohol, and rinse again with distilled water. Dry after each rinsing by wiping lightly with optical lens tissue.

Washington, D.C. 20234
January 8, 1980

George A. Uriano, Chief
Office of Standard Reference Materials

(over)



Spectral transmittance of a typical didymium glass filter. Numbers indicate the principal points of minimum transmittance and letters indicate the principal points of inflection.

TABLE 1

SRM 2014
FILTER D3
Wavelengths (nm) of the transmittance minima
for the indicated bandwidths.

BANDWIDTH	1.5NM	3.0NM	4.5NM	6.0NM	7.5NM	9.0NM	10.5NM
MINIMUM NO.							
1	402.35	401.68	401.49	401.36	400.98	400.24	
2	431.49	432.45					
3	440.16	440.39	441.57	442.39	442.22	441.89	441.03
4	445.63	445.17					
5	472.70	472.57	472.93				
6	478.80	479.25	479.20	478.26	477.35	476.51	475.66
7	513.42	513.59	513.87	514.30	515.39		
8	529.63	530.10	529.99	529.53	529.31	529.15	528.91
9	572.67	573.23	574.17	575.05	576.50		
10	585.31	585.52	585.76	586.02	585.99	585.36	584.43
11	623.63	624.03					
12	629.42	629.38	628.51	626.97	626.98		
13	684.67	684.70	684.74	684.75	684.73	684.68	684.60
14	739.90	739.96	740.24	740.90	741.99	742.94	743.63
15	748.27	748.08					

Table 2

Wavelengths and Transmittances at Nine Selected Points of Inflection

Point Identification	Wavelength (nm)	Range* (nm)	Transmittance†
A	406.38	+.08 −.06	0.7562
B	429.42	+.05 −.05	.7293
C	449.45	+.06 −.06	.6514
D	484.77	+.10 −.15	.6743
E	536.52	+.06 −.09	.5801
F	568.16	+.08 −.04	.4008
G	598.99	+.05 −.07	.3346
H	733.45	+.06 −.03	.4730
I	756.48	+.01 −.02	.4215

*The range of wavelengths within which the wavelength for the given transmittance will fall for symmetric triangular passbands with half-height bandwidths from 1.5 to 10.5 nm.

†These values of transmittance are not certified.

SRM 2014

Table 3

Estimated Random and Systematic Errors of the Transmittance Minima

Band Number	Nominal Wavelength of Minimum Transmittance	Standard Deviation for Indicated Bandwidth						
		1.5 nm	3.0 nm	4.5 nm	6.0 nm	7.5 nm	9.0 nm	10.5 nm
1	402 nm	0.015 nm (0.07)†	0.018 nm	0.020 nm	0.021 nm	0.025 nm	0.029 nm	–
2	431	0.013 (0.06)	0.010	–	–	–	–	–
3	440	0.021 (0.05)	0.013	0.050	0.028	0.009	0.005	0.007 nm (0.25)
4	446	0.023 (0.06)	0.029	–	–	–	–	–
5	473	0.011 (0.08)	0.012	0.032	–	–	–	–
6	479	0.015 (0.15)	0.013	0.009	0.009	0.009	0.014	0.017 (0.25)
7	513	0.022 (0.08)	0.016	0.014	0.013	0.010	–	–
8	530	0.012 (0.21)	0.010	0.010	0.011	0.010	0.011	0.010 (0.25)
9	573	0.004 (0.05)	0.010	0.010	0.012	0.014	–	–
10	585	0.007 (0.06)	0.004	0.007	0.008	0.008	0.007	0.007 (0.25)
11	624	0.058 (0.06)	0.061	–	–	–	–	–
12	630	0.210 (0.02)	0.120	0.171	0.133	0.091	–	–
13	685	0.019 (0.05)	0.029	0.024	0.017	0.014	0.014	0.012 (0.25)
14	740	0.009 (0.25)	0.013	0.011	0.010	0.009	0.009	0.010 (0.25)
15	748	0.020 (0.04)	0.016	–	–	–	–	–

†Values in parentheses are estimates of the systematic error. The estimates for the 1.5 nm bandwidth were obtained from the data taken at 1.5 nm intervals as compared to data taken at 0.15 nm intervals. The method of estimating the systematic errors for the 10.5 nm bandwidth is described in Section 3.4 in SP 260-66.

SRM 2014

TABLE 4

Transmittance (T) as a function of wavelength from 380 to 780.5 nm for a bandwidth of 1.5 nm. The estimates of random and systematic errors are also provided. These data are not certified.

SRM 2014
FILTER D3

WAVE-LENGTH (NM)	T	ΔT STANDARD ERROR	ΔT SYSTEMATIC	WAVE-LENGTH (NM)	T	ΔT STANDARD ERROR	ΔT SYSTEMATIC
380.00	.56408	.000166	.00020	381.50	.58927	.000158	.00020
383.00	.61549	.000224	.00020	384.50	.63913	.000168	.00020
386.00	.66074	.000036	.00020	387.50	.68019	.000148	.00020
389.00	.70000	.000200	.00020	390.50	.71596	.000148	.00020
392.00	.73059	.000562	.00020	393.50	.74159	.000126	.00020
395.00	.74908	.000371	.00020	396.50	.74695	.000431	.00020
398.00	.72948	.000147	.00020	399.50	.70711	.000209	.00020
401.00	.69490	.000428	.00020	402.50	.68362	.000087	.00020
404.00	.72360	.000381	.00020	405.50	.74684	.000293	.00020
407.00	.76517	.000232	.00020	408.50	.79399	.000365	.00020
410.00	.80951	.000448	.00020	411.50	.81618	.000042	.00020
413.00	.82014	.000004	.00020	414.50	.82279	.000420	.00020
416.00	.82273	.000202	.00020	417.50	.82355	.000148	.00020
419.00	.82484	.000191	.00020	420.50	.83133	.000174	.00020
422.00	.83041	.000209	.00020	423.50	.82769	.000173	.00020
425.00	.82973	.000208	.00020	426.50	.82916	.000229	.00020
428.00	.81332	.000144	.00020	429.50	.73002	.000429	.00020
431.00	.62838	.000289	.00020	432.50	.64134	.000161	.00020
434.00	.64801	.000081	.00020	435.50	.64135	.000088	.00020
437.00	.62778	.000179	.00020	438.50	.61194	.000166	.00020
440.00	.60400	.000144	.00020	441.50	.60792	.000154	.00020
443.00	.61344	.000095	.00020	444.50	.60859	.000283	.00020
446.00	.60560	.000168	.00020	447.50	.61992	.000148	.00020
449.00	.64707	.000144	.00020	450.50	.67304	.000572	.00020
452.00	.68938	.000140	.00020	453.50	.69950	.000185	.00020
455.00	.71018	.000580	.00020	456.50	.71102	.000112	.00020
458.00	.69851	.000144	.00020	459.50	.67728	.000053	.00020
461.00	.65876	.000163	.00020	462.50	.65011	.000161	.00020
464.00	.64916	.000003	.00020	465.50	.64446	.000051	.00020
467.00	.63254	.000032	.00020	468.50	.61760	.000171	.00020
470.00	.60380	.000282	.00020	471.50	.58545	.000072	.00020
473.00	.57581	.000167	.00020	474.50	.59748	.000313	.00020
476.00	.60693	.000204	.00020	477.50	.57178	.000297	.00020
479.00	.56222	.000147	.00020	480.50	.57066	.000315	.00020
482.00	.58791	.000180	.00020	483.50	.63290	.000305	.00020
485.00	.68802	.000153	.00020	486.50	.72915	.000144	.00020
488.00	.76019	.000431	.00020	489.50	.78756	.000137	.00020
491.00	.80846	.000352	.00020	492.50	.82013	.000444	.00020
494.00	.82381	.000270	.00020	495.50	.82173	.000711	.00020
497.00	.81535	.000042	.00020	498.50	.80481	.000137	.00020

TABLE 4 (cont)

SRM 2014
FILTER D3

WAVE-LENGTH (NM)	T	ΔT STANDARD ERROR	ΔT SYSTEM-ATIC	WAVE-LENGTH (NM)	T	ΔT STANDARD ERROR	ΔT SYSTEM-ATIC
500.00	.79105	.000313	.00020	501.50	.77626	.000087	.00020
503.00	.76044	.000093	.00020	504.50	.74601	.000161	.00020
506.00	.72982	.000217	.00020	507.50	.70548	.000281	.00020
509.00	.66187	.000082	.00020	510.50	.60232	.000136	.00020
512.00	.55353	.000145	.00020	513.50	.53338	.000143	.00020
515.00	.55132	.000286	.00020	516.50	.58027	.000172	.00020
518.00	.59881	.000076	.00020	519.50	.60934	.000074	.00020
521.00	.59245	.000190	.00020	522.50	.52976	.000028	.00020
524.00	.45433	.000157	.00020	525.50	.42574	.000054	.00020
527.00	.41618	.000045	.00020	528.50	.38625	.000279	.00020
530.00	.37984	.000030	.00020	531.50	.38911	.000143	.00020
533.00	.40520	.000141	.00020	534.50	.46553	.000144	.00020
536.00	.55842	.000071	.00020	537.50	.64441	.000079	.00020
539.00	.70722	.000036	.00020	540.50	.75790	.000133	.00020
542.00	.79718	.000308	.00020	543.50	.82391	.000293	.00020
545.00	.84216	.000171	.00020	546.50	.85455	.000277	.00020
548.00	.86261	.000159	.00020	549.50	.86652	.000148	.00020
551.00	.86644	.000284	.00020	552.50	.86644	.000206	.00020
554.00	.86582	.000283	.00020	555.50	.86479	.000147	.00020
557.00	.86138	.000284	.00020	558.50	.85472	.000169	.00020
560.00	.84407	.000178	.00020	561.50	.82791	.000141	.00020
563.00	.80051	.000158	.00020	564.50	.75648	.000177	.00020
566.00	.67427	.000053	.00020	567.50	.51342	.000165	.00020
569.00	.26737	.000287	.00020	570.50	.08434	.000010	.00020
572.00	.03012	.000007	.00020	573.50	.02986	.000009	.00020
575.00	.04042	.000006	.00020	576.50	.04508	.000132	.00020
578.00	.05932	.000139	.00020	579.50	.07972	.000139	.00020
581.00	.07928	.000270	.00020	582.50	.04534	.000138	.00020
584.00	.01892	.000002	.00020	585.50	.01290	.000138	.00020
587.00	.01969	.000007	.00020	588.50	.03571	.000138	.00020
590.00	.05516	.000140	.00020	591.50	.06625	.000001	.00020
593.00	.08585	.000011	.00020	594.50	.13210	.000007	.00020
596.00	.20756	.000283	.00020	597.50	.28554	.000140	.00020
599.00	.34273	.000058	.00020	600.50	.40063	.000052	.00020
602.00	.46448	.000280	.00020	603.50	.52727	.000164	.00020
605.00	.57996	.000323	.00020	606.50	.61507	.000051	.00020
608.00	.63507	.000143	.00020	609.50	.65418	.000081	.00020
611.00	.68840	.000165	.00020	612.50	.74187	.000092	.00020
614.00	.80164	.000424	.00020	615.50	.84914	.000364	.00020
617.00	.87574	.000173	.00020	618.50	.88587	.000286	.00020

TABLE 4 (cont)

SRM 2014
FILTER D3

WAVE-LENGTH (NM)	T	ΔT STANDARD ERROR	ΔT SYSTEM-ATIC	WAVE-LENGTH (NM)	T	ΔT STANDARD ERROR	ΔT SYSTEM-ATIC
620.00	.88508	.000108	.00020	621.50	.87833	.000314	.00020
623.00	.87244	.000286	.00020	624.50	.87272	.000450	.00020
626.00	.87509	.000308	.00020	627.50	.87493	.000172	.00020
629.00	.87194	.000240	.00020	630.50	.87289	.000199	.00020
632.00	.87728	.000156	.00020	633.50	.88332	.000157	.00020
635.00	.88925	.000174	.00020	636.50	.89184	.000282	.00020
638.00	.89372	.000343	.00020	639.50	.89566	.000559	.00020
641.00	.89773	.000417	.00020	642.50	.89921	.000618	.00020
644.00	.90012	.000439	.00020	645.50	.90091	.000147	.00020
647.00	.90140	.000330	.00020	648.50	.90113	.000066	.00020
650.00	.90110	.000168	.00020	651.50	.90090	.000177	.00020
653.00	.90175	.000226	.00020	654.50	.90259	.000243	.00020
656.00	.90292	.000075	.00020	657.50	.90232	.000589	.00020
659.00	.90232	.000419	.00020	660.50	.90101	.000321	.00020
662.00	.89970	.000340	.00020	663.50	.89692	.000181	.00020
665.00	.89216	.000163	.00020	666.50	.88622	.000207	.00020
668.00	.87926	.000281	.00020	669.50	.87307	.000167	.00020
671.00	.86880	.000240	.00020	672.50	.86491	.000147	.00020
674.00	.86150	.000174	.00020	675.50	.85653	.000179	.00020
677.00	.84784	.000419	.00020	678.50	.83564	.000442	.00020
680.00	.82112	.000148	.00020	681.50	.80418	.000717	.00020
683.00	.78494	.000624	.00020	684.50	.77042	.000831	.00020
686.00	.77968	.000551	.00020	687.50	.79835	.000179	.00020
689.00	.81423	.000237	.00020	690.50	.83138	.000582	.00020
692.00	.84679	.000583	.00020	693.50	.86246	.000265	.00020
695.00	.87290	.000451	.00020	696.50	.88178	.000314	.00020
698.00	.88812	.000377	.00020	699.50	.89313	.000211	.00020
701.00	.89612	.000738	.00020	702.50	.89793	.000246	.00020
704.00	.90071	.000287	.00020	705.50	.90138	.000238	.00020
707.00	.90137	.000342	.00020	708.50	.90141	.001539	.00020
710.00	.90232	.000587	.00020	711.50	.90148	.000626	.00020
713.00	.90076	.000325	.00020	714.50	.89856	.000303	.00020
716.00	.89591	.000490	.00020	717.50	.89302	.000725	.00020
719.00	.88801	.000596	.00020	720.50	.88303	.000733	.00020
722.00	.87502	.000302	.00020	723.50	.86449	.000290	.00020
725.00	.85057	.000207	.00020	726.50	.83083	.000568	.00020
728.00	.79900	.000638	.00020	729.50	.75293	.000443	.00020
731.00	.68007	.000207	.00020	732.50	.56964	.000448	.00020
734.00	.42722	.000283	.00020	735.50	.28729	.000281	.00020
737.00	.19664	.000288	.00020	738.50	.16498	.000140	.00020

TABLE 4 (cont)

SRM 2014
FILTER D3

WAVE-LENGTH (NM)	T	ΔT STANDARD ERROR	ΔT SYSTEM-ATIC	WAVE-LENGTH (NM)	T	ΔT STANDARD ERROR	ΔT SYSTEM-ATIC
740.00	.16030	.000148	.00020	741.50	.16548	.000137	.00020
743.00	.18770	.000565	.00020	744.50	.21318	.000296	.00020
746.00	.21285	.000145	.00020	747.50	.19637	.000283	.00020
749.00	.19647	.000144	.00020	750.50	.22309	.000148	.00020
752.00	.26453	.000139	.00020	753.50	.31332	.000169	.00020
755.00	.36843	.000151	.00020	756.50	.42998	.000425	.00020
758.00	.49007	.000140	.00020	759.50	.54324	.000140	.00020
761.00	.58938	.000172	.00020	762.50	.62755	.000474	.00020
764.00	.66334	.000105	.00020	765.50	.69617	.000171	.00020
767.00	.72629	.000162	.00020	768.50	.75118	.000288	.00020
770.00	.77113	.000584	.00020	771.50	.78394	.000140	.00020
773.00	.79334	.000289	.00020	774.50	.79545	.000463	.00020
776.00	.79344	.000764	.00020	777.50	.78342	.000858	.00020
779.00	.76568	.000268	.00020	780.50	.73923	.000091	.00020

U.S. Department of Commerce
Elliot L. Richardson,
Secretary

National Bureau of Standards
Ernest Ambler, Acting Director

National Bureau of Standards Certificate

Standard Reference Material 2030

Glass Filter for Transmittance Measurement

R. Mavrodineanu and J. R. Baldwin

Set # 70

This Standard Reference Material is intended as a reference source for one-point verification of the transmittance and absorbance scales of spectrophotometers at the given wavelength and measured transmittance. It consists of one glass filter in its holder and one empty filter holder. The filter bears an identification number. For protection, the metal holder is provided with two shutters that should be removed during measurements.

The transmittance, T, at λ 465.0 nm and for a spectral bandpass of 2.7 nm is: 0.3168. The corresponding transmission density, $-\log_{10}T$, is: 0.4992.

Date of Certification: January 1, 1981.

The transmittance value (T) can be converted to percent transmittance (%T) by multiplying by 100. The transmission density is calculated from the measured transmittance (T). This value should be indicated by the absorbance scale of the spectrophotometer if the filter is measured against air.

The transmittance value given was measured against air at an ambient temperature of 23.5 °C, and is estimated to be accurate to within ±0.5 percent at the time of certification. This uncertainty includes the random and systematic errors of the calibration procedure, as well as transmittance changes of the filter during the period of calibration.

It is possible that aging of the glass may cause some filters to change transmittance by about ±1 percent over a period of approximately 1 year from the date of calibration. Improper storage or handling of the filter may also cause changes [5]. In cases where verification is desirable, the filter should be returned to the National Bureau of Standards for cleaning and recalibration.

It is recommended that the filter in the holder be handled only by the edges and with soft plastic (polyethylene) gloves and optical lens tissue. When not in use, it should be stored in its holder, with the shutters on, and in the box provided for this purpose. Extended exposure to laboratory atmosphere and dusty surroundings should be avoided.

This Standard Reference Material was issued with the assistance and advice of K. D. Mielenz and J. R. DeVoe.

The technical and support aspect involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. W. Seward.

Washington, D.C. 20234
September 23, 1976

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

The transmittance measurements were made using the high-accuracy spectrophotometer designed and built at the Institute for Materials Research, National Bureau of Standards [1]. This instrument represents the primary transmittance standard; its transmittance accuracy was established using the double-aperture method of linearity testing [1,3,5,6]. The reproducibility (relative standard deviation obtained from a set of 20 measurements) of this high-accuracy spectrometer for transmittance measurements on glass filters similar to the one used to produce this SRM, is approximately 0.02 percent.

The combined uncorrected systematic error of the instrument is estimated to be not more than $\pm 10^{-4}$ transmittance units.

As indicated on the face of this certificate, SRM 2030 is intended to be used as a one-point verification of the transmittance (absorbance) scales of spectrophotometers. If these scales require verification at other wavelengths and transmittances, "Glass Filters for Spectrophotometry," SRM 930c or subsequent issues, should be used.

The neutral glass for the filter was provided by Schott of Mainz, Germany, and is designated as "Jena Color and Filter Glass " [2,5].

The exposed surface of the glass is approximately 29 × 8 mm, measuring from a point 1.5 mm above the base of the filter holder (see figure). The empty filter holder is provided to be used in the reference beam of the spectrophotometer so that approximately equivalent conditions of stray radiations are achieved for both beams.

The transmittance of the filter depends upon the intrinsic properties of the material, spectral bandpass, wavelength, geometry of the optical beam, temperature, and positioning of the filter. A change of ambient temperature of 1 or 2 °C from 23.5 °C will not significantly affect the calibration [5].Changes in the transmittance may be caused by surface conditions, aging of the glass, exposure to a harmful atmosphere, or careless handling as indicated on the face of this certificate [2,3,4,5]. The effective spectral bandpass used to determine the certified value is given on the face of the certificate and the transmittance measurements are made by producing the vertical image of the slit (about 8 mm by 0.5 mm), using a convergent beam geometry with an aperture ratio f:10, in the middle of the entrance face of the glass filter. The filter was measured in the spectrophotometer in a position perpendicular to the incident light beam as shown in the attached figure.

Prior to certification measurements, the filter was examined for surface defects and thoroughly cleaned [5]. Should the surface of the glass filter become contaminated, no attempt should be made to clean it unless the user has the facilities to demonstrate that the cleaning treatment has not altered the glass surface or degraded the accuracy of the certified value. Because the Standard Reference Material is a transfer standard, the only means available to verify its integrity is remeasuring its transmittance with a primary standard instrument similar to that used in this certification [1,4,5]. In most cases where verification, or recertification, of the transmittance value is desirable, it will be most expeditious to return the filter to the National Bureau of Standards for measurement. Prior to shipment the NBS Office of Standard Reference Materials should be contacted regarding the conditions and cost of verification and/or recertification.

REFERENCES

[1] R. Mavrodineanu, An Accurate Spectrophotometer for Measuring the Transmittance of Solid and Liquid Materials, NBS Journal of Research **76A**, No. 5, 405-425 (1972).

[2] R. Mavrodineanu, Solid Materials to Check the Photometric Scale of Spectrophotometers, NBS Tech. Note 544, O. Menis and J. I. Shultz, ed., pp 6-17, U.S. Government Printing Office, Washington, D.C. 20402 (Sept. 1970), ibid NBS Tech. Note 584, pp 2-21 (December 1971).

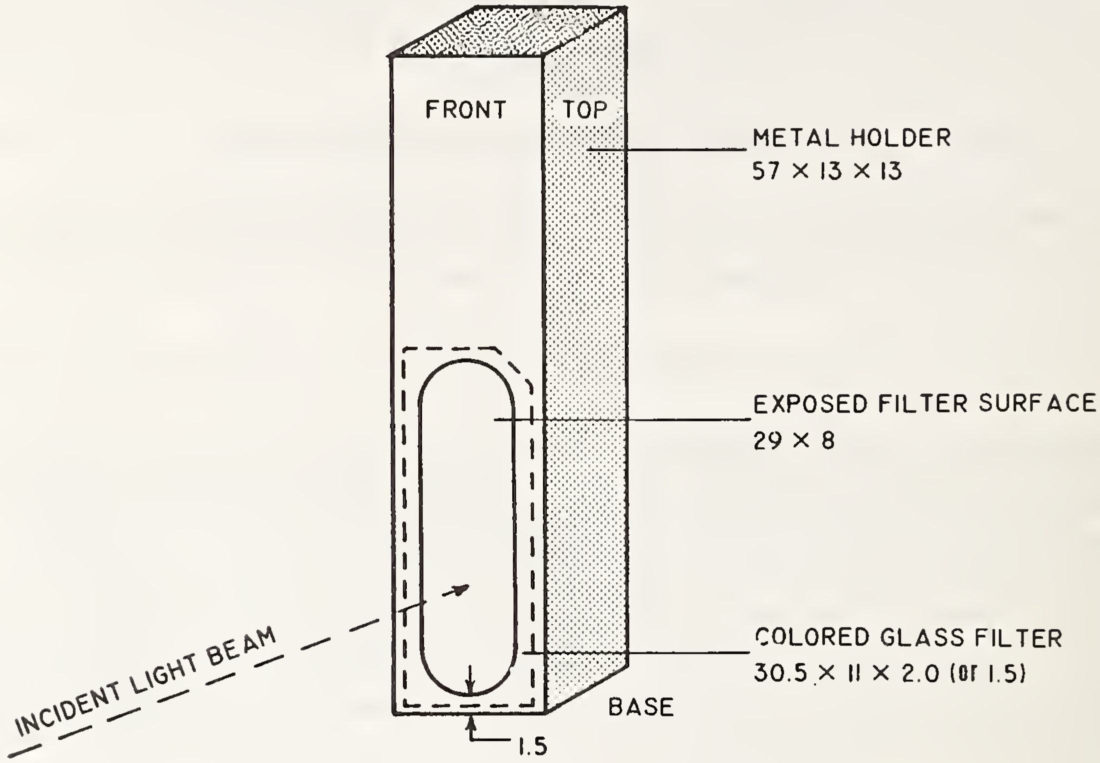
[3] K. S. Gibson, Spectrophotometry, NBS Circ. 484 (Sept. 1949).

[4] Collected Papers from NBS Conference on Accuracy in Spectrophotometry and Luminescence Measurements, NBS Journal of Research **76A**, No. 5, 375-510 (1972).

[5] R. Mavrodineanu and J. R. Baldwin, Glass Filters as a Standard Reference Material for Spectrophotometry-Selection, Preparation, Certification, Use SRM 930, NBS Special Publication 260-51, U.S. Government Printing Office, Washington, D.C. 20402, (1975).

[6] K. D. Mielenz and K. L. Eckerle, Spectrophotometric Linearity Testing Using Double-Beam-Aperture Method, Appl. Optics, **11**, 2294-2303 (1972).

METAL HOLDER FOR THE COLORED GLASS FILTERS



DIMENSIONS IN mm

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards Certificate

Standard Reference Material 2031

Metal-on-Quartz Filters for Spectrophotometry

R. Mavrodineanu and J. R. Baldwin

This Standard Reference Material is intended for use in the verification of the transmittance and absorbance scales of spectrophotometers in the ultraviolet and visible spectral regions. It consists of three individual filters in their metal holders and one empty filter holder.

Each filter is made from two non-fluorescent fused silica plates, one of which carries a semi-transparent evaporated chromium metal layer while the other is clear. The two plates comprising the filter are assembled by optical contact. The chromium coating on two of the filters produces nominal transmittances of 10 percent and 30 percent. The third filter is made from assembling two clear fused silica plates by optical contact, and has a nominal transmittance of 90 percent. The metal holders for these filters are provided with shutters to protect the filters when not in use. These shutters must be removed at the time of measurement and be replaced after the measurements have been completed. Each filter bears an identification number.

Filter & Set Ident. Number	TRANSMITTANCE (T)									
	Wavelength, nm									
	250.0	280.0	340.0	360.0	400.0	465.0	500.0	546.1	590.0	635.0
20–10	0.1232	0.1176	0.1085	0.1087	0.1069	0.0990	0.1004	0.1083	0.1179	0.1284
20–30	0.4314	0.4176	0.3809	0.3736	0.3593	0.3267	0.3185	0.3206	0.3311	0.3432
20–90	0.9132	0.9203	0.9273	0.9282	0.9301	0.9323	0.9330	0.9334	0.9338	0.9343

Filter & Set Ident. Number	TRANSMITTANCE DENSITY ($-\log_{10}T$)									
	Wavelength, nm									
	250.0	280.0	340.0	360.0	400.0	465.0	500.0	546.1	590.0	635.0
20–10	0.909	0.930	0.965	0.964	0.971	1.004	0.998	0.965	0.928	0.891
20–30	0.3651	0.3792	0.4192	0.4276	0.4445	0.4859	0.4969	0.4940	0.4800	0.4645
20–90	0.0394	0.0361	0.0328	0.0324	0.0315	0.0304	0.0301	0.0299	0.297	0.0295

Date of Certification: January 1, 1981

Washington, D.C. 20234
June 1, 1979

George A. Uriano, Chief
Office of Standard Reference Materials

(over)

The transmittance values (T) can be converted to percent transmittance (%T) by multiplying by 100. The transmittance densities are calculated from the measured transmittance (T). The transmittance densities should be indicated by the absorbance scale of the spectrophotometer if the filters are measured against air. The transmittance values given were measured against air at an ambient temperature of 23.5 °C.

The transmittance values for the filter with a nominal transmittance of 10 percent are estimated to be accurate to within ± 1.0 percent at the time of certification. The transmittance values for the filters having a nominal transmittance of 30 percent and 90 percent are estimated to be accurate to within ± 0.5 percent at the time of certification. This uncertainty includes the effects of random and systematic errors of the calibration procedure, as well as possible transmittance changes of the filters during the period of calibration.

Aging of the material may cause some filters to change transmittance with time. Improper storage or accidental touching of the filter surface may also cause changes.

When not in use, the filters should be stored in their holders with the shutters in place and in the metal container provided for this purpose. Extended exposure to laboratory atmosphere and dirty surroundings should be avoided. In cases where verification is desirable, the filters should be returned to the National Bureau of Standards for cleaning and recalibration. It is advisable to perform the verification on a yearly basis.

The transmittance measurements were made using the high-accuracy spectrophotometer designed and built at the NBS Center for Analytical Chemistry [1]. This instrument represents the primary transmittance standard; its transmittance accuracy was established using the double-aperture method of linearity testing [1,3,5,6]. The estimated standard deviation obtained from a set of 20 measurements of the high-accuracy spectrophotometer used for transmittance measurements on filters of the SRM 2031 type is 5×10^{-5} transmittance units for transmittance between 0.1 and 0.3. The bounds of the systematic error of the instrument are estimated to be $\pm 10^{-4}$ transmittance units.

The transmittance, T, was measured against air in the reference beam; hence it includes the reflection losses that occur at the air-filter interface. Under these circumstances the measured transmittance, T, has a corresponding transmittance density, defined as $-\log_{10} T$. The internal transmittance, T_i, of a material is defined as the transmittance of the material corrected for reflection losses. This is obtained experimentally when the measurements are made against a blank sample in the reference beam. The absorbance, A, of a material is related to this internal transmittance, T_i, by the expression, $A = -\log_{10} T_i$. The transmittance measurements are made by producing the vertical image of the slit (about 8 mm by 0.5 mm), using a convergent beam geometry with an aperture ratio f:10, in the middle of the entrance face of the filter. The filters were measured in the spectrophotometer in a position perpendicular to the incident light beam.

The metal-on-quartz filters which constitute this SRM were produced according to NBS specifications by Cosmo Optics, Inc., Middletown, New York. The quartz plates were produced from non-fluorescent fused silica material of optical quality, ground and polished to produce an optical contact. The metal used to produce, by evaporation,the semi-transparent metal coating was chromium, obtained through the van Arkel process, and has an indicated purity of 99.998 percent. It was provided by The Atomergic Chemetals Co., Plainview, New York. The fused silica plates were ground and polished at the same time and on the same grinding wheel, each to the nominal dimensions: 1.5 mm thick, 30.5 mm long, and 10.4 mm wide.

The exposed surface of each filter is approximately 29 x 8 mm, measuring from a point 1.5 mm above the base of the filter holder (see figure). The empty filter holder provided is to be used in the reference beam of the spectrophotometer so that approximately equivalent conditions of stray radiation are achieved for both beams. The transmittance of the filters depends upon the intrinsic properties of the material, wavelength, spectral bandpass, geometry of the optical beam, temperature, and positioning of the filter. A change of ambient temperature of 1 or 2 °C from 23.5 °C will not significantly affect the calibration [5]. Changes in the transmittance may be caused by surface conditions, aging of the material, exposure to a harmful atmosphere, or careless handling [2,3,4,5].

SRM 2031 is stored in a black anodized aluminum container provided with a threaded cap made of the same metal. Each filter is placed in a cylindrical cavity to prevent any contact between the filter face and the walls of the storage container. Contamination of the filter surface with particulate matter due to static charges is minimized through the metallic nature of the container. A flat leaf spring is inserted into the cylindrical cavity with each filter holder to minimize damage during transportation. These springs can be removed during normal use in the laboratory.

The filter is shown in the assembled unit with its front surface facing up. The filter, in its filter holder, should be placed in the cuvette compartment of the spectrophotometer with its front surface facing the incident light beam and the rear surface facing the photodetector.

Top: Cylindrical container with its screw cap, both made of black-anodized aluminum alloy. Four filter holders can be stored in the cylindrical container. Bottom (from left to right): Aluminum alloy filter holder, 12.5 mm square and 58 mm high; Metal-on-quartz filter; Retaining spring of beryllium-copper with nylon screw and washer; Assembled unit; and Two Delrin shutters. All metal and plastic parts are flat black.

Prior to certification measurements, each filter was examined for surface defects and the condition of the optical contact [5]. Should the surface of the filter become contaminated, no attempt should be made to clean it unless the user has the facilities to demonstrate that the cleaning treatment will not alter the surface or degrade the accuracy of the certified values. As SRM 2031 is a transfer standard, the only means available to verify its integrity is to remeasure its transmittance with a primary standard instrument similar to that used in this certification [1,5]. In most cases, where verification or recertification of the transmittance values is desirable, it will be most expeditious to return the filters to the National Bureau of Standards for measurement. Prior to shipment, the NBS Office of Standard Reference Materials should be contacted regarding the conditions and cost of verification and/or recertification.

Further information concerning the selection, preparation and properties of SRM 2031 will be found in reference 5.

This Standard Reference Material was issued with the assistance and advice of K.D. Mielenz and I.L. Barnes.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.W. Seward.

We wish to acknowledge the cooperation of George N. Bowers, Jr., M.D., of Hartford Hospital, Hartford, Connecticut; Royden N. Rand, Ph.D., of the Eastman Kodak Co. Research Laboratories, Rochester, New York; and Donald S. Young, M.D., Ph.D., of the Mayo Clinic, Rochester, Minnesota.

The contributions of H.E. Bennett and J.M. Bennett of the Michelson Laboratory, Physical Optics Branch, Naval Weapons Center, China Lake, California, who helped in the production of SRM 2031 are also gratefully acknowledged.

References

1. R. Mavrodineanu, An Accurate Spectrophotometer for Measuring the Transmittance of Solid and Liquid Materials, NBS Journal of Research **76A**, No. 5, 405-425 (1972).
2. R. Mavrodineanu, Solid Materials to Check the Photometric Scale of Spectrophotometers, NBS Tech. Note 544, O. Menis and J.I. Shultz, Eds., pp. 6-17, U.S. Government Printing Office, Washington, D.C. 20402 (Sept. 1970); ibid, NBS Tech. Note 584, pp. 2-21 (December 1971).
3. K.S. Gibson, Spectrophotometry, NBS Circ. 484 (Sept. 1949).
4. R. Mavrodineanu, Considerations for the Use of Semi-transparent Metallic Thin Films as Potential Transmittance Standards in Spectrophotometry. NBS Journal of Research **80A**, No. 4, 637-641 (1976).
5. R. Mavrodineanu and J.R. Baldwin, Metal-on-Quartz Filters as a Standard Reference Material for Spectrophotometry, SRM 2031. NBS Special Publication 260-68, U.S. Government Printing Office, Washington, D.C. 20402, 1979.
6. K.D. Mielenz and K.L. Eckerle, Spectrophotometer Linearity Testing Using the Double-Aperture Method, Appl. Optics **11**, 2294-2303 (1972).

U. S. Department of Commerce
Philip M. Klutznick
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards
Certificate

Standard Reference Material 2033

Crystalline Potassium Iodide with Attenuator
Heterochromatic and Isochromatic Stray Radiant Energy Standard for Ultraviolet Absorption Spectrophotometry

K. D. Mielenz and R. Mavrodineanu

This Standard Reference Material consists of reagent-grade crystalline potassium iodide (KI) and a radiation attenuator to be used to assess the heterochromatic stray radiant energy (stray light) in ultraviolet absorption spectrophotometers in the spectral region below 260 nm.. The radiation attenuator consists of two semitransparent evaporated metal-on-fused silica (non-fluorescent) filters, each having a nominal transmittance of 10%. One filter is mounted in a cuvette-style holder that can be inserted into the sample compartment of the spectrophotometer. The other is mounted in one of the two shutters of the holder. The use of this attenuator permits the expansion of the transmittance scale into the low-transmittance region by providing a two-step attenuation of the reference beam of the spectrophotometer to about 1%. The attenuator can also be used to assess isochromatic stray light by following the instructions given in this certificate.

The heterochromatic stray light is assessed by measuring the spectral absorbance of aqueous solutions of SRM 2033 of known pathlengths and concentrations, and comparing the results with the certified values of the specific absorbance, ϵ.

Specific Absorbance[a]

	ϵ (L g^{-1} cm^{-1}), vs Wavelength, λ (nm), at 23.5 °C							
λ	240	245	250	255	260	265	270	275
ϵ	25.6	10.67	3.66	1.05	0.260	0.0560	0.0121	0.0031

[a]"Specific Absorbance" is defined here as absorbance per unit pathlength and unit concentration. The term "absorptivity" has been avoided since it is ambiguously defined. See K. D. Mielenz, Anal. Chem. 48, 1093-1094 (1976).

The estimated uncertainty of these values is ± 5%, which includes the random and systematic errors of the calibration procedure, as well as variations due to possible instability of the KI. (See Certification Procedure.)

The material used to produce SRM 2033 was obtained from the J.T. Baker Chemical Co., Phillipsburg, N.J., in two bottles, as reagent-grade (99.8% purity) potassium iodide.

The transmittance of the filter mounted in the holder at λ255 nm is ____________;
the transmittance of both filters at λ255 is ____________.

SRM 2033 was issued with the technical assistance of J. R. Baldwin, R. W. Burke, A. L. Cummings, B. I. Diamondstone, and G. A. Sleater, and under the overall direction of E. L. Garner, NBS Inorganic Analytical Research Division.

The technical and support aspects concerning preparation, certification, and issuance of this SRM were coordinated through the Office of Standard Reference Materials by R. W. Seward.

Washington, D.C. 20234
May 9, 1980

George A. Uriano, Chief
Office of Standard Reference Materials

(over)

Potassium Iodide Material Testing:

Material homogeneity was tested by measuring the absorbance of 1% aqueous solutions of the KI sampled from the top, middle, and bottom of each bottle. These measurements, at 265, 267, and 270 nm, showed no evidence of inhomogeneity. Tests for moisture content (Karl Fischer method) gave an average of 0.007%. This value was sufficiently small that all certified data are based on weighed samples of the undried, as received, material.

Stability of the material to UV and visible light was tested by exposing the KI salt in a low-actinic glass bottle to radiation from a 3 kW xenon-arc lamp at a distance of 25 cm for four days at room temperature. The average absorbances at five wavelengths between 240 and 270 nm of aqueous solutions of the exposed material were 1.1% lower than those of solutions of the unexposed material. In view of the severity of this test, this change was considered minor. Nonetheless, the 1.1% was included in the overall uncertainty of the certified values.

Potassium Iodide Certification Procedure:

The certified specific absorbances were measured in the NBS Center for Analytical Chemistry high-accuracy spectrophotometer [1,2], equipped with a deuterium lamp and UV averaging sphere [3,4]. Thirteen aqueous solutions of KI with concentrations ranging from 0.03 to 30 g L^{-1} were prepared and measured against distilled water, using standard fused-silica cuvettes with pathlengths between 10.001 and 10.009 mm[b]. The absorbance measurements were performed at the temperature, t = 23.5±0.5 °C. A 0.2 nm bandpass was used, and wavelength settings were made with an accuracy of 0.05 nm. The measurements were made on at least three concentrations at each wavelength. These concentrations were chosen so that most absorbance readings fell between 0.1 and 1.0 to minimize stray light and bandwidth errors. The data were found to obey Beer's law.

Thus, the equation,

$$\epsilon = A/c\ell \tag{1}$$

was used to calculate the specific absorbances, ϵ, from the measured absorbances, A, and the known values of concentration, c, and pathlength, ℓ .

Limits to random error (two standard deviations) of these measurements (including the photometric imprecision of the spectrophotometer, errors in the repositioning of cuvettes, concentration errors, pathlength uncertainties, temperature errors, Beer's law uncertainties, and bandwidth errors) were determined to be ± 2.4%. The error due to the 0.05 nm uncertainty of the wavelength calibration of the spectrophotometer is ± 1.1%.

The uncertainty of 5% stated on the face of this certificate represents the sum of these errors and the above mentioned 1.1% uncertainty due to instability of the KI. No corrections were applied for the effects of internal reflections inside the sample and reference cuvettes, nor were the weights corrected to vacuum. These and all other sources of error were considered negligible.

The temperature coefficient at 260 nm was determined to be

$$\frac{1}{\epsilon} \cdot \frac{d\epsilon}{dt} = 0.031\ {}^{\circ}C^{-1}. \tag{2}$$

For precision measurements, it is recommended that SRM 2033 be used in thermostated cuvettes at 23.5 °C, or that a temperature correction according to Eq. (2) be applied.

[b]Issued by NBS as SRM 932, Quartz Cuvette for Spectrophotometry.

Instructions for Use of Potassium Iodide

Storage and Preparation:

SRM 2033 should be stored in the original, low-actinic glass bottle and the cardboard container in which it was issued to protect it from unnecessary exposure to light and humidity. When so stored, the expected stability of this material is at least three years. Thus, until additional stability data are obtained, this material should be used within three years of the date of purchase.

All solutions prepared from SRM 2033 should be made in borosilicate glass containers using distilled water and transfer pipettes (Pasteur type) of the same glass and fitted with rubber bulbs[c]. Use clean spectrophotometer cuvettes, free of scratches, made of non-fluorescent fused silica, and fitted with ground-glass or Teflon stoppers to minimize evaporation; or preferably use NBS SRM 932. Mark all cuvettes to assure the same orientation in the spectrophotometer, and place them into their respective holders. Using transfer pipettes of the type mentiond, rinse each cuvette several times with distilled water. Prepare a solution of KI in distilled water (e.g., 1%), fill the sample cuvette with the KI solution and the reference cuvette with distilled water. Measure the absorbance, leaving the cuvettes in their holders, empty them (using the pipettes), and repeat the rinsing and filling operations until constant absorbance readings are obtained. Fresh solutions should be made before every test.

Measurements:

A 1% KI solution ($c = 10\ g\ L^{-1}$) with a 1 cm pathlength exhibits a sharp cutoff in transmittance near 260 nm; i.e., it transmits more than 90% above 273 nm, but less than 0.01% below 258 nm. Therefore, with the monochromator set for a wavelength below 260 nm, any appreciable amount of light detected is heterochromatic stray light, which consists of wavelengths above the cutoff. The amount of stray light in the spectrophotometer at wavelength λ may be determined from the equations:

$$T'(\lambda) = \frac{T(\lambda) + x(\lambda)}{1 + x(\lambda)} \tag{3}$$

$$x(\lambda) = \frac{T'(\lambda) - T(\lambda)}{1 - T'(\lambda)} \tag{4}$$

where:

$x(\lambda)$ is the stray light ratio,

$T'(\lambda)$ is the apparent transmittance,

$T(\lambda)$ is the true transmittance.

The stray light ratio, $x(\lambda)$, is the proportion of heterochromatic stray radiant energy in the spectrophotometer for the wavelength setting λ. Equation (4) forms the theoretical basis for the determination of the stray light ratio by comparing apparent transmittance to true transmittance. For SRM 2033, the true transmittance, $T(\lambda)$, of KI solutions may be calculated as:

$$T(\lambda) = 10^{-\epsilon(\lambda)\, c\, \ell}$$

where $\epsilon(\lambda)$ is the certified specific absorbance given on the face of this certificate.

[c] Soft glass containers and pipettes contain residual amounts of UV absorbing material, but can be used after proper cleaning. Several rinses, first with isopropyl alcohol and then with distilled water, are generally adequate.

The apparent transmittances of KI solutions depend not only on the stray light ratio of the spectrophotometer, but also on the wavelength accuracy and the spectral bandwidth, which can significantly affect the results obtained because of the steep slope of the absorbance cutoff of KI. A low-pressure mercury discharge lamp is suggested for verifying the wavelength scale, and a bandwidth as narrow as compatible with adequate signal-to-noise ratios should be used.

Optical Attenuator:

Many instruments do not permit the direct measurement of transmittances below certain limits, e.g., 1% or 0.1%. To measure stray light ratios below these limits, the reference beam of the spectrophotometer must be attenuated to extend the transmittance scale into the low-transmittance region.

As mentioned on the face of this Certificate, the optical attenuator consists of two filters, each of which has a nominal transmittance of 10 percent. One filter is mounted in a metal holder that can be inserted in the sample compartment of the spectrophotometer (5). This holder is provided with a front and rear shutter, one of which has a window in which the second filter is mounted. The holder and shutters are flat black. This unit was produced in the NBS Instrument Shops.

Instructions for Use of the Attenuator

This unit can be used to attenuate the incident radiation in the reference beam of the spectrophotometer in two steps by a total factor of about 100, corresponding to about 1 percent transmittance (6). To attenuate by a factor of 10, the attenuator is inserted in the sample compartment of the spectrophotometer facing the incident beam, with both shutters removed. A further attenuation by a factor of about 10 is obtained when the shutter carrying the second filter is inserted in the holder, in front of the first filter. Under these conditions, and with an instrument that cannot measure transmittances lower than 1 percent, the use of the attenuator will permit heterochromatic stray light to be measured down to about 0.01 percent. This attenuation procedure can be used with spectrophotometers capable of scale expansion.

Isochromatic Stray Light:

The radiation attenuator can be used to assess the isochromatic stray light that results from reflection of the incident radiation at the surface of the sample and various optical components, and reaches the photodetector without passing through the sample. It is implicitly assumed that the sample compartment of the spectrophotometer is light-tight.

The measurement is performed by placing the attenuator in the sample beam of the spectrophotometer, with the opaque shutter placed at the rear of the filter holder. Under these conditions, if a signal is detected, it is caused by reflection at the surface of the filter exposed to the incident radiation. This radiation is scattered from the walls and other components of the sample compartment and reaches the photodetector without passing through the sample (5). This signal is the isochromatic stray light.

Isochromatic stray light, which passes through the sample, is generally caused by interreflections between lenses and/or other sample compartment elements. Tests for this stray light component, which is not detected by the methods described above, are discussed in references 7 and 8.

References:

1. R. Mavrodineanu, J. Res. NBS 76A, 405-425 (1972).
2. J. F. Barkley, F. C. Ruegg, and R. Mavrodineanu, 29th Pittsburgh Conf., Abstract 452, Cleveland, Ohio, (1978).
3. K. D. Mielenz, R. Mavrodineanu, and E. D. Cehelnik, J. Res. NBS 78A, 631-635 (1974).
4. K. D. Mielenz, R. Mavrodineanu, and E. D. Cehelnik, Appl. Optics 14, 1940-1947 (1975).
5. R. Mavrodineanu, J. Res. NBS 80A, 637-641 (1976).
6. Estimating Stray Radiant Energy, ASTM Manual on Recommended Practices in Spectrophotometry, pp. 94-105, 3rd. Edition, 1969, 1916 Race St., Philadelphia, Pa. 19103.
7. K. D. Mielenz, J. Res. NBS 76A, 455-467 (1972).
8. K. D. Mielenz and R. Mavrodineanu, J. Res. NBS 77A, 699-703 (1973).

U. S. Department of Commerce
Frederick B. Dent
Secretary
National Bureau of Standards
Richard W. Roberts, Director

National Bureau of Standards
Certificate
Standard Reference Material 2203
Potassium Fluoride
(Standard for Ion-Selective Electrodes)

E. S. Etz

This Standard Reference Material, certified for the activity of fluoride ion in solutions of this salt, is made available for the standardization of fluoride ion-selective electrodes. The material is of analytical reagent-grade purity, but should not be considered entirely free of traces of chloride, fluosilicates and heavy metals. Potentiometric measurements on solutions of this material employing fluoride ion-selective electrodes in cells without liquid junction indicate this material to be homogeneous within the precision (± 0.003 pF unit) of the measured potential of the fluoride ion-selective electrode. Chemical analysis of this material indicates a minimum assay of 99.5 percent KF after drying.

This material is certified for the activity coefficient of fluoride ion and the related value of pF ($\equiv -\log a_{F^-}$, where a_{F^-} is a conventional activity of fluoride ion) at 25 °C for solutions of potassium fluoride over the range of molalities m = 0.0001 to m = 2.0. Certified values of a_{F^-} and pF(S) at selected molalities are found in the table on the reverse page. The pF(S) values given are estimated to be accurate to ± 0.01 pF unit.

The mean molal activity coefficient of potassium fluoride from m = 0.0001 to m = 2.0 may be represented at 25 °C by the equation:

$$\log \gamma_{\pm} = \frac{-Am^{1/2}}{1+Bm^{1/2}} + \beta m + Cm^2$$

where m is the molality. The numerical values of the constants appearing in this expression are, at 25 °C:

$$A = 0.5108$$
$$B = 1.2880$$
$$\beta = 2.7843 \times 10^{-2}$$
$$C = 4.6933 \times 10^{-3}$$

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of R. A. Durst.

The technical and support aspects involved in the preparation, certification, and issuance of this standard reference material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D. C. 20234
May 21, 1973

J. Paul Cali, Chief
Office of Standard Reference Materials

Values of $\gamma_{\pm}$ calculated from the relation given above represent the most reliably known experimental activity coefficient data to an uncertainty of no larger than ± 0.005 in the value of $\gamma_{\pm}$ over the range of molalities indicated.

Certified Values of Ionic Activity (a_{F^-}) Single Ion Activity

Coefficients (γ_{F^-}) and Standard Values of

pF ($\equiv -\log a_{F^-}$) for Fluoride Ion at 25 °C

(all referred to the molality scale)

KF Molality (m)	Molarity (c)	Fluoride Ion Concentration* (g/l)	Activity Coefficient $\gamma_{F^-} = \gamma_{K^+} = \gamma_{\pm}$	Activity $a_{F^-} = a_{K^+} = a_{\pm}$	pF (S)
0.0001	0.0000997	0.00189	0.988	0.0000988	4.00_5
0.0005	0.000499	0.00947	0.975	0.000487	3.31_2
0.001	0.000997	0.0189	0.965	0.000965	3.01_6
0.005	0.00498	0.0947	0.927	0.00464	2.33_4
0.01	0.00997	0.1893	0.902	0.00902	2.04_5
0.05	0.0498	0.9463	0.818	0.0409	1.38_8
0.1	0.0996	1.892	0.773	0.0773	1.11_2
0.2	0.1990	3.781	0.726	0.145	0.83_8
0.3	0.2982	5.665	0.699	0.210	0.67_8
0.5	0.4961	9.425	0.670	0.335	0.47_5
0.75	0.7424	14.104	0.652	0.489	0.31_1
1.0	0.9873	18.757	0.645	0.645	0.19_0
1.5	1.4729	27.983	0.646	0.969	$+0.01_4$
2.0	1.9523	37.091	0.658	1.316	-0.11_9

*To convert to parts per million (mg/l) multiply by 10^3

The equation relating the mean molal activity coefficient to molality may be used to calculate values of pF at concentrations other than those appearing in the table. The convention used here for the assignment of ionic activities in solutions of potassium fluoride is based on hydration theory and is described by Robinson, Duer and Bates [Anal. Chem. 43, 1862 (1971)]. For this particular 1:1 electrolyte, the convention leads to $\gamma_{F^-} = \gamma_{K^+}$ in aqueous solution, and values of a_{F^-} are therefore obtained by the relation

$$a_{F^-} = m\gamma_{F^-} = m\gamma_{\pm}$$

Preparation of Standard Solutions

To prepare a 1.0 molal solution, transfer 57.363 g of potassium fluoride (weight in air) to a 1-liter volumetric flask. Dissolve and fill to the mark with distilled water at 25 °C. The distilled water should have a conductivity no greater than $2 \times 10^{-6}\,\Omega^{-1}$. The potassium fluoride is hygroscopic and should be dried in two consecutive stages; initially for a period of 2 hours at 110 °C, followed by drying at 200 ± 10 °C for an additional 2 hours. After drying, normal precautions should be exercised to prevent pickup of moisture, i.e., minimal exposure to humid conditions prior to and during weighing.

Similarly, a 0.1 molal solution may be prepared by the transfer of 5.786 g of potassium fluoride (weight in air) to a 1-liter volumetric flask, dissolving, and diluting to the mark with distilled water at 25 °C. Appropriate dilution of either standard solution should be used to obtain standards in the concentration range of interest to the user.

After preparation, standard solutions should be transferred to non-silicate storage containers, e.g., polyethylene bottles, to avoid fluosilicate formation and the resulting decrease in fluoride ion activity.

Electrode Calibration

In order to minimize residual liquid junction potential errors arising at the reference electrode junction, it is recommended that reference standards be used at concentrations similar to those of the samples. Also, the use of a bracketing technique, whereby two standard solutions bracket the concentration range of the samples, will increase the reliability of the measurements by minimizing errors due to non-Nernstian electrode response.

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards
Certificate
Standard Reference Material 2032
Crystalline Potassium Iodide
Heterochromatic Stray Radiant Energy Standard for Ultraviolet Absorption Spectrophotometry

K. D. Mielenz and R. Mavrodineanu

This Standard Reference Material consists of reagent-grade, crystalline potassium iodide (KI) to be used to assess heterochromatic stray radiant energy (stray light) in ultraviolet absorption spectrophotometers in the spectral region below 260 nm. Stray light is assessed by measuring the spectral absorbance of aqueous solutions of SRM 2032 of known pathlengths and concentrations, and comparing the result with the certified values of the specific absorbance, ϵ.

Specific Absorbance[a]

ϵ (L g^{-1} cm^{-1}), vs Wavelength, λ (nm), at 23.5 °C

λ	240	245	250	255	260	265	270	275
ϵ	25.6	10.67	3.66	1.05	0.260	0.0560	0.0121	0.0031

[a] "Specific absorbance" is defined here as absorbance per unit pathlength and unit concentration. The term "absorptivity" has been avoided since it is ambiguously defined. See K. D. Mielenz, Anal. Chem. 48, 1093-1094 (1976).

The estimated uncertainty of these values is ± 5%, which includes the random and systematic errors of the calibration procedure, as well as variations due to possible instability of the KI. (See, Certification Procedure.)

The material used to produce SRM 2032 was obtained from the J. T. Baker Chemical Co., Phillipsburg, N.J., in two bottles, as reagent-grade (99.8% purity) potassium iodide.

SRM 2032 was issued with the technical assistance of J. R. Baldwin, R. W. Burke, A. L. Cummings, B. I. Diamondstone, and G. A. Sleater, and under the overall direction of I. L. Barnes.

The technical and support aspects concerning preparation, certification, and issuance of this SRM were coordinated through the Office of Standard Reference Materials by R. W. Seward.

Washington, D.C. 20234
October 31, 1979

George A. Uriano, Chief
Office of Standard Reference Materials

(over)

Supplementary Information

Material Testing:

Material homogeneity was tested by measuring the absorbance of 1% aqueous solutions of the KI sampled from the top, middle, and bottom of each bottle. These measurements, at 265, 267, and 270 nm, showed no evidence of inhomogeneity. Tests for moisture content (Karl Fischer method) gave an average of 0.007%. This value was sufficiently small that all certified data are based on weighed samples of the undried, as received, material.

Stability of the material to UV and visible light was tested by exposing the KI salt in a low-actinic glass bottle to radiation from a 3 kW xenon-arc lamp at a distance of 25 cm for four days at room temperature. The average absorbances at five wavelengths between 240 and 270 nm of aqueous solutions of the exposed material were 1.1% lower than those of solutions of the unexposed material. In view of the severity of this test, this change was considered minor. Nonetheless, the 1.1% was included in the overall uncertainty of the certified values.

Certification Procedure:

The certified specific absorbances were measured in the NBS Center for Analytical Chemistry high-accuracy spectrophotometer [1, 2], equipped with a deuterium lamp and UV averaging sphere [3, 4]. Thirteen aqueous solutions of KI with concentrations ranging from 0.03 to 30 g L^{-1} were prepared and measured against distilled water, using standard fused-silica cuvettes with pathlengths between 10.001 and 10.009 mm[b]. The absorbance measurements were performed at the temperature, t = 23.5 ± 0.5 °C. A 0.2 nm bandpass was used, and wavelength settings were made with an accuracy of 0.05 nm. The measurements were made on at least three concentrations at each wavelength. These concentrations were chosen so that most absorbance readings fell between 0.1 and 1.0 to minimize stray light and bandwidth errors. The data were found to obey Beer's law.

Thus, the equation,

$$\epsilon = A/c\,\ell \qquad (1)$$

was used to calculate the specific absorbances, ϵ, from the measured absorbances, A, and the known values of concentration, c, and pathlength, ℓ .

Limits to random error (two standard deviations) of these measurements (including the photometric imprecision of the spectrophotometer, errors in the repositioning of cuvettes, concentration errors, pathlength uncertainties, temperature errors, Beer's law uncertainties, and bandwidth errors) were determined to be ± 2.4%. The error due to the 0.05 nm uncertainty of the wavelength calibration of the spectrophotometer is ± 1.1%.

The uncertainty of 5% stated on the face of this certificate represents the sum of these errors and the above mentioned 1.1% uncertainty due to instability of the KI. No corrections were applied for the effects of internal reflections inside the sample and reference cuvettes, nor were the weights corrected to vacuum. These and all other sources of error were considered negligible.

The temperature coefficient at 260 nm was determined to be

$$\frac{1}{\epsilon}\frac{d\epsilon}{dt} = 0.031\ °C^{-1}. \qquad (2)$$

For precision measurements, it is recommended that SRM 2032 be used in thermostated cuvettes at 23.5 °C, or that a temperature correction according to Eq. (2) be applied.

[b]Issued by NBS as SRM 932, Quartz Cuvettes for Spectrophotometry.

Instructions for Use

Storage and Preparation:

SRM 2032 should be stored in the original, low-actinic glass bottle and the cardboard container in which it was issued to protect it from unnecessary exposure to light and humidity. When so stored, the expected stability of this material is at least three years. Thus, until additional stability data are obtained, this material should be used within three years of the date of purchase.

All solutions prepared from SRM 2032 should be made in borosilicate glass containers using distilled water and transfer pipettes (Pasteur type) of the same glass and fitted with rubber bulbs[c]. Use clean spectrophotometer cuvettes, free of scratches, made of non-fluorescent fused silica, and fitted with ground-glass or Teflon stoppers to minimize evaporation; or preferably use NBS SRM 932. Mark all cuvettes to assure the same orientation in the spectrophotometer, and place them into their respective holders. Using transfer pipettes of the type mentioned, rinse each cuvette several times with distilled water. Prepare a solution of KI in distilled water (e.g., 1%), fill the sample cuvette with the KI solution and the reference cuvette with distilled water. Measure the absorbance, leaving the cuvettes in their holders, empty them (using the pipettes), and repeat the rinsing and filling operations until constant absorbance readings are obtained. Fresh solutions should be made before every test.

Measurements:

A 1% KI solution ($c = 10\ g\ L^{-1}$) with a 1 cm pathlength exhibits a sharp cutoff in transmittance near 260 nm; i.e., it transmits more than 90% above 273 nm, but less than 0.01% below 258 nm. Therefore, with the monochromator set for a wavelength below 260 nm, any appreciable amount of light detected is heterochromatic stray light, which consists of wavelengths above the cutoff. The amount of stray light in the spectrophotometer at wavelength λ may be determined from the equations:

$$T'(\lambda) = \frac{T(\lambda) + x(\lambda)}{1 + x(\lambda)} \qquad (3)$$

$$x(\lambda) = \frac{T'(\lambda) - T(\lambda)}{1 - T'(\lambda)} \qquad (4)$$

where:

$x(\lambda)$ is the stray light ratio,

$T'(\lambda)$ is the apparent transmittance,

$T(\lambda)$ is the true transmittance.

The stray light ratio, $x(\lambda)$, is the proportion of heterochromatic stray radiant energy in the spectrophotometer for the wavelength setting λ. Equation (4) forms the theoretical basis for the determination of the stray light ratio by comparing apparent transmittance to true transmittance. For SRM 2032, the true transmittance, $T(\lambda)$, of KI solutions may be calculated as:

$$T(\lambda) = 10^{-\epsilon(\lambda)\, c\, \ell}$$

where $\epsilon(\lambda)$ is the certified specific absorbance given on the face of this certificate.

[c]Soft glass containers and pipettes contain residual amounts of UV absorbing material, but can be used after proper cleaning. Several rinses, first with isopropyl alcohol and then with distilled water, are generally adequate.

The apparent transmittances of KI solutions depend not only on the stray light ratio of the spectrophotometer, but also on the wavelength accuracy and the spectral bandwidth, which can significantly affect the results obtained because of the steep slope of the absorbance cutoff of KI. A low-pressure mercury discharge lamp is suggested for verifying the wavelength scale, and a bandwidth as narrow as compatible with adequate signal-to-noise ratios should be used.

Many instruments do not permit the direct measurement of transmittances below certain limits, e.g., 1% or 0.1%. For such instruments, the use of SRM 2032 will only measure stray light ratios above these limits. To measure stray light ratios below these limits, the reference beam of the spectrophotometer must be attenuated to extend the transmittance scale into the low-transmittance region.

References

1. R. Mavrodineanu, J. Res. NBS **76A**, 405-425 (1972).

2. J.F. Barkley, F. C. Ruegg, and R. Mavrodineanu, 29th Pittsburgh Conf., Abstract 452, Cleveland, Ohio, (1978).

3. K. D. Mielenz, R. Mavrodineanu, and E. D. Cehelnik, J. Res. NBS **78A**, 631-635 (1974).

4. K. D. Mielenz, R. Mavrodineanu, and E. D. Cehelnik, Appl. Optics **14**, 1940-1947 (1975).

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R.W. Seward and R. Mavrodineanu

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11. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)*

This publication is a summary of the Clinical Laboratory Standards issued by NBS as Standard Reference Materials (SRM's). The material, composition, certification, use, and remarks concerning each of the SRM's described are presented in tabular form. Copies of the certificates for these SRM's are contained in the appendix for more detailed information.

12. KEY WORDS *(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)*

Chemical composition; clinical materials; Standard Reference Materials

13. AVAILABILITY

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PERIODICALS

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NOTE: The Journal was formerly published in two sections: Section A "Physics and Chemistry" and Section B "Mathematical Sciences."

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NOTE: The principal publication outlet for the foregoing data is the Journal of Physical and Chemical Reference Data (JPCRD) published quarterly for NBS by the American Chemical Society (ACS) and the American Institute of Physics (AIP). Subscriptions, reprints, and supplements available from ACS, 1155 Sixteenth St., NW, Washington, DC 20056.

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